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INNOVATIVE CLEAN TECHNOLOGIES CASE STUDIES SECOND YEAR PROJECT REPORT

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This study was conducted in cooperation with the USEPA Office of Small and Disadvantaged Business Utilization

RISK REDUCTION ENGINEERING LABORATORY OFFICE OF RESEARCH AND DEVELOPMENT U.S. ENVIRONMENTAL PROTECTION AGENCY CINCINNATI, OH 45268



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FOREWORD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of materials that, if improperly dealt with, can threaten both public health and the environment. The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. These laws direct EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Risk Reduction Engineering Laboratory is responsible for planning, implementing, and managing research, development and demonstration programs to provide and authoritative, defensible engineering basis in support of the policies, programs, and regulations of the EPA with respect to drinking water, wastewater, pesticides, toxic substances, solid and hazardous wastes, and Superfund-related activities. This publication is one of the products of that research and provides a vital communication link between the researcher and the user community.

This report summarizes projects supported by the RREL to promote the development of innovative pollution prevention technologies and techniques by small businesses. The reader is encouraged to contact the individual developers listed in each summary for more information regarding their process or product.

E. Timothy Oppelt, Director Risk Reduction Engineering Laboratory

ABSTRACT

The Innovative Clean Technologies Case Studies contained herein are the products of the "Pollution Prevention by and for Small Business" Program (P2SB). The P2SB was an outreach program directed to small businesses that had developed innovative concepts for pollution prevention in their industries. The P2SB focused on high-risk concepts without emphasis on media or industry in order to provide an open program where ground-breaking concepts were given a fair opportunity.

The P2SB provided awards of up to \$25,000 to assist small businesses for conducting their own demonstrations of pollution prevention techniques and technologies, and for advancing their products towards a practical stage.

In its second year, the P2SB funded projects in a variety of industries across the nation. This publication provides a history of the P2SB and lists case histories of the projects funded in the second year. A earlier report entitled "Inovative Clean Technologies Case Studies" (EPA/600/R-93/175) covering the first year of the program is already available.

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ACKNOWLEDGEMENTS

The Office of Research and Development has supported the P2SB in cooperation with the Office of Small and Disadvantaged Business Utilization and is grateful for the efforts of Karen V. Brown, EPA's Small Business Ombudsman, who and coordinated support for this program. Through this office the support of the following trade associations is acknowledged:

National Small Business United Small Business Legislative Council National Automobile Dealers Association Independent Lubricant Manufacturers Association American Wood Preservers Institute Institute of Industrial Launderers National Tooling and Machining Association Printing Industries of America, Inc. International Fabricare Institute Synthetic Organic Chemical Manufacturers Association Chemical Specialties Manufacturers Association National Association of Metal Finishers Chemical Producers and Distributors Association National Federation of Independent Businesses National Fertilizer Solutions Association National Association of Truck Stop Operators Society of Plastics Industry, Inc. Graphic Arts Technical Foundation American Galvanizers Association

The support to small businesses provided by Angel Martin-Dias at the Center for Hazardous Materials Research is appreciated. The chapters covering individual case histories were written by Ms. Martin-Dias, and CHMR retains copies of individual reports. Support for the commercialization of selected technologies, which was beyond the scope of P2SB, was provided by National Environmental Technology Applications Center (NETAC). The advice and expertise provided by the American Institute for Pollution Prevention (AIPP) was invaluable to the selection process.

This report was compiled and finalized by Dr. Rada Olbina. Her efforts are greatly appreciated.

INTRODUCTION

In 1989, the U.S. Environmental Protection Agency established a 2% Set-aside Program to fund pollution prevention initiatives from across the Agency. These set-asides were instituted to encourage the research, development and demonstration of pollution prevention concepts, techniques and technologies nationwide.

One such initiative, Pollution Prevention by and for Small Business (P2SB), was proposed by the Office of Small and Disadvantaged Business Utilization (OSDBU) with the support of the Office of Research and Development (ORD). This initiative was selected for funding under the pollution prevention 2% Set-aside, with co-funding provided by ORD. The P2SB was managed through a cooperative agreement with the Center for Hazardous Materials Research (CHMR), and some of the P2SB small businesses received additional support for the commercialization of their technologies through the National Environmental Technology Applications Center (NETAC). Nineteen trade associations supported the program though promotion, advise and information transfer.

The P2SB provided awards of up to \$25,000 to assist small businesses for conducting their own demonstrations of pollution prevention techniques and technologies, and for advancing their concepts to a practical stage. The P2SB was a three-year program, ending September 1993, with awards being made in the 1991 and 1992 Fiscal Years of the Federal Government. This publication covers those projects completed from the 1992 year, and represents the completation of this program.

The P2SB small business applied their own knowledge and expertise in the field to structure their projects and data collection activities in a manner they determined would provide the most effective means of furthering the development of their concepts. The reader should be aware that the data provided in these summaries are not the results of third party evaluations.

The in-house demonstrations were completed in 14-16 months and reports filed with CHMR which in turn developed research briefs for Agency review. These research briefs have been re-organized into the chapters of this publication. The technologies are considered promising research and development concepts, and while several have advanced towards commercialization, others require further investigation and testing. All are presented to provide the reader the opportunity to contact the small business demonstrator for potential uses.

The success of the P2SB program depends on the involvement of the trade associations who sponsor presentations by the participating small businesses at annual conferences and regional workshops. This initiative was developed to support critical pollution prevention efforts in a variety of facilities and industries that might not otherwise have the chance or the resources to reduce wastes or to test and implement their innovative ideas. The P2SB was an attempt to support promising ventures and encourage further development. It also expanded EPA's knowledge of pollution prevention needs in different sectors, supporting government, business and public cooperation in finding ways to prevent pollution.

PHASE II AND III VENTED TWO STAGE VALVES FOR INTERNAL COMBUSTION ENGINES

by

Reggie D. Huff Acro-Tech, Inc. Tigard, OR 97223

ABSTRACT

Acro-Tech, Inc.'s Vented Two-stage Valving project assessed a concept for improving the induction process of the internal combustion engine. The duty of the induction cycle is to move air and fuel molecules from an inlet venturi to a cylindér while creating enough turbulence to break up the fuel molecules so they can be burned efficiently. Unlike standard popet valves, which are of a one piece, one function design, vented valves are of a two piece, two function design. This design facilitates a reduction in mechanical stress and an increased efficiency in air flow dynamics. The net results are expected to be improved engine performance, decreased emissions, and enhanced fuel efficiency by creating a more dilute, homogeneous induction charge, and by allowing better control of induction process timing.

INTRODUCTION

PROJECT DESCRIPTION

Valve Technology Background

An internal combustion engine can be described as a sophisticated air pump that must be able to take in large amounts of air (intake), mix that air with fuel (usually in liquid form, but sometimes in gaseous form, such as propane) at precise ratios, continue to move the air/fuel mixture into a combustion chamber, seal itself completely, compress the mixture, ignite it, thoroughly extract the energy released through the resulting expansion of the mixture, and completely purge itself of the spent gases. This process must occur in a fraction of a second. At an extremely modest operating speed of 2,000 revolutions per minute (RPM), this entire process must take place within six hundredths of a second or less, and each one of the four main cycles described has to complete its job efficiently in less than fifteen thousandths of a second.

The induction process, which occurs in the first cycle of operation, is the key to all four cycles working harmoniously and efficiently. The duty of the induction cycle is to move air and fuel molecules from an inlet venturi to a cylinder within a very short time; at just the right time; and to fill the cylinder while creating enough turbulence to break up the fuel molecules to allow efficient combustion.

The intake valve is always located at the cylinder end of the induction airflow pattern. Low pressure is created in the cylinder when the piston, which runs within the cylinder, begins to descend rapidly down the cylinder on the intake stroke. The opening of the intake valve must be timed precisely, opening as expeditiously as possible, staying open for just the right amount of time, and closing as fast as mechanically possible. Then, the valve must seal completely and be able to withstand tremendous heat and pressure.

Unfortunately, even though the intake valve's job is to allow the induction system to flow as freely as possible within a predetermined time sequence, the head of the valve itself is the most detrimental obstruction to the air flow pattern.

Basic Flow Dynamics of Vented Valves

Unlike standard popet valves, which are of a one piece, one function design, vented valves are of a two piece, two function design. The main, or *outer valve*, controls the flow of the port and is very similar in size and shape to a standard valve, but is designed with vents in the head portion. This facilitates the need for a much smaller *inner valve* to control the flow through the vents, as shown in Figures 1 and 2. This concept allows for a reduction in mechanical stress.

Figures 1a and 1b. Standard Intake Valve Flow Dynamics

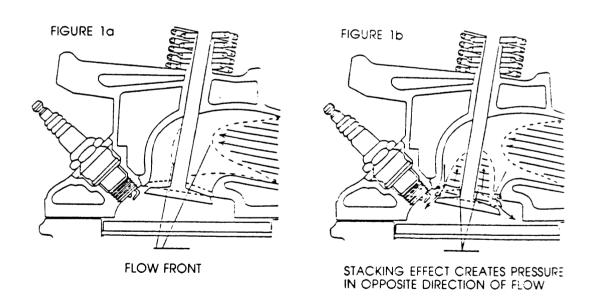
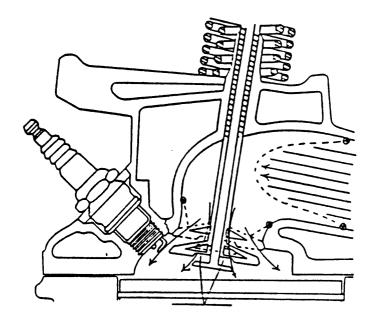
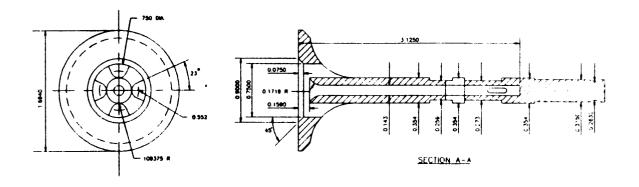


Figure 2. Vented Intake Valve Flow Dynamics



LOW PRESSURE CREATED IN CENTER OF VALVE RELIEVES STACKING EFFECT

Figure 3. Vented Valve Prototype



3

The vented valve concept follows the basic law of flow dynamics: the further a gas or liquid flows away from its given boundaries, the more efficiently it will flow within those boundaries. As the inner valve opens, the column of air in the intake port begins to collapse inward through the series of vents, past the inner valve, and into the combustion chamber. The center 30 to 50 percent of the air column moves first, giving laminar flow a more direct passage into the combustion chamber. This alleviates the need for the center of the air column to be radically diverted around the head of the valve and into the same space where the outer boundary of the air column is flowing. After the center of the air column is moving, the outer valve is opened, giving direct flow passage to the outer 50 to 70 percent column of air. This same operation continues in reverse as the valve mechanism closes.

The standard valve design attempts to create air flow that is opposite of normal flow dynamics by moving the outer flow first and preventing the inner laminar flow from moving faster and more efficiently through the port.

Whenever the natural flow pattern of the port is changed, much of the flow dimension is lost and the performance potential of the entire engine is greatly affected (such as with a standard valve). The vented valve creates a flow that is directly in line with natural flow dynamics by creating a multi-layered pathway that taps into higher velocity, more efficient areas of the port exit.

The Dilute Homogeneous Air and Fuel Combustion Process

A homogeneous air/fuel mixture is obtained when fuel molecules are completely separated and distributed evenly through a given air mass. Unfortunately, fuel molecules have a tendency to cling together, forming droplets and pools that burn and release heat very slowly, as compared to completely atomized fuel. Since the combustion process must occur in such a short time, a slow combustion process means that fuel may be unused, unburned, to be expelled into the atmosphere and deteriorating air quality.

Vented valve technology enhances pre-combustion turbulence and creates a more homogeneous air/fuel mixture. When the inner valve mechanism is first actuated, an extreme low pressure point is created in the center of the outer valve that is surrounded by higher pressure. A natural phenomenon, the vortex effect, occurs, and is much like pulling the plug in a bathtub, only more violent. This forces the center column of air/fuel mixture to move first so it will be continually fed through the vents in a high speed swirling action throughout the operation of the valve mechanism. Creating turbulence directly at and past the intake valve operation is one of the most appropriate ways of creating a turbulent, homogeneous air-diluted fuel charge, because this event is well-timed with the combustion process. Vented valves tap directly into the high velocity center area of laminar flow, allowing and even promoting its high velocity characteristics, rather than acting as a brake or perturbation to it. All flow is forced through the center vents at two points in the operation of the vented valve, promoting high velocity. Therefore, a new kinetic value is seen at the valve, and there is more force, or energy to be diffused into turbulence as it enters the combustion chamber instead of creating a high pressure area above the combustion chamber. This is the only way that turbulence can be increased while maintaining or improving flow dimension.

Some may feel that today's fuel injectors homogenize the fuel molecules sufficiently such that the aforementioned turbulence is not relevant. Even with throttle body injectors or central fuel injection, one can observe the speed with which the finely sprayed fuel molecules pool together and stream down the throttle body walls. If the injectors are placed directly in front of the intake valve, their value as homogenizers is increased; however, the example given shows the value of turbulence, no matter how or when fuel is introduced into the system.

Several advantages may be realized by breaking fuel droplets apart by means of turbulence just before combustion. Improved flame propagation and velocity allows the combustion process to take place more quickly. This allows for improved combustion timing to promote performance. More of the combustion process can be utilized before the exhaust valve opens, which can improve emission quality dramatically.

Complex Flow Dynamics of Vented Valves

By reviewing Figures 1 and 2, one can obtain a clearer understanding of how the vented valve concept functions in an internal combustion engine.

If a dormant intake port instantly begins to fill with air/fuel mixture from top to bottom, induced by either thrust pressure or vacuum pressure as the valve is actuated, then the shape of the flow front that would occur instantly could be illustrated as shown in Figure 1a; the center area pushes ahead of the outer area, flowing several times faster and more efficiently than the trailing edge of the flow front. A parabolic lobe-shaped flow front would appear. This condition would occur even with the use of micropolished surfaces. In following the flow front down the port and closer to the standard intake valve, one can note that the most efficient area of flow (i.e., the center of the flow front lobe) will contact the valve base at the center. therefore having the longest distance to travel along the base of the valve before entering the combustion chamber. This condition is detrimental to efficient center flow, so that when the flow front makes contact with the base of the valve (Figure 1b), it begins to flatten while waiting to enter the combustion chamber. When considering that the flow front is actually a continuous wave, a stacking effect can occur on top of the valve base, as high pressure is created in the center area of the valve. This stacking effect pushes the high pressure lobe in the exact opposite direction of the flow. This provides another opportunity for even atomized fuel molecules to pool together.

Although the standard valve design has served well in terms of simplicity, reliability, and serviceability, it poses the greatest liability to the efficiency of the entire induction system, even if the system includes a turbo or super charger. The standard valve design is simply not allowed enough time to optimize the flow around itself and continues to deteriorate in efficiency as engine RPM increases. The vented valve shows a marked improvement, allowing for more flexibility in cam profiles with less overlap for lower emissions and better fuel efficiency, a broader torque and power band, as well as increased maximum power and torque output levels, before considering the merit of the forcefully induced swirl effect.

Acro-Tech identifies "cycle-to-cycle flow kinetics" as a defining factor that determines the torque and power curve of a given engine. Cycle-to-cycle flow kinetics is based conversely on Newton's Law of Motion. Thus, the greater the speed of motion, the greater the outside force needed to slow that motion. For example, a standard valve engine allows a certain velocity of flow in cycle A. The kinetic energy stored in that flow will determine how long the flow will continue to move through the port after the valve has closed on cycle A. This determines how high the pressure or charge behind the valve will be when the valve opens for cycle B, and so on, until the outside dynamic forces determine the maximum velocity allowed. Components, such as tunnel rams, increase cycle-to-cycle kinetic energy simply because it takes more energy, or outside force, to stop the movement of a tall column of air than a short one. Therefore, the air charge is packed tightly behind the valve after every cycle, propagating velocity. By allowing the very important center column of air to move faster and more efficiently through the port, vented valves will increase cycle-to-cycle kinetic energy as well.

APPLICATION

Valve Replacement Benefits

Since vented valves are designed to replace components within the same operational space, they would be very cost effective to install, as there would be few redesign requirements. The cost of the vented valve component will be higher than a traditional valve because it is slightly more complex, has more moving parts, and is fabricated from more costly materials such as stainless steel, titanium, and molybdenum. New technology is generally expensive; however cost increases in one area can sometimes be offset by the cost savings in another area.

Enhanced performance, fuel efficiency, and emission quality realized through the use of vented valves could allow the auto manufacturer to reduce costs in several areas. With improved performance, it may be possible to replace 4 valve-per-cylinder engines with 2 valve engines, thus reducing the number of parts used and simplifying production. The use of down-sized versions of existing engine designs may also be possible. Improved fuel efficiency can save petroleum products. Better emissions could reduce the need for expensive emission control equipment necessary to meet stringent emission standards. For example, catalytic converters, containing costly platinum as a catalyst, are used in exhaust systems to burn-off unburned hydrocarbons leaving the engine. An improvement in pre-catalyst exhaust could result in a simpler catalytic converter.

Cross Segment Uses

Overall market potential for vented valve technology is quite large and diverse. The twelve main market segments, in no significant order, are:

- <u>Automotive</u>: original equipment manufacturers (OEM)
- Truck: light, heavy duty, semi (OEM)
- Industrial: machine power, generators, etc. (OEM)
- Engine rebuilders: auto, truck, etc., aftermarket manufacturers (AM)
- Engine modifiers: high performance, racing (AM)

Defense

- Government: federal, state, local fleet, etc.
- Aviation: (OEM), (AM)Marine: (OEM), (AM)
- Recreational vehicles: motorcycles, etc. (OEM), (AM)
- Agriculture: (OEM), (AM)
- Locomotive

The automotive and light and heavy duty truck segments have the most to gain from successful development of vented valve technology. However, the cost of implementing new technology is relative to the benefit gained. Clear 10 percent improvements in fuel efficiency, emissions, and performance may be worth a cost increase of 20 percent. However, a 1 percent improvement might be considered to expensive at a 5 percent increase in costs.

PROCEDURE

DEMONSTRATION SET-UP

The engine selected for the project was a 1989 Mitsubishi 2.0 liter, 4 cylinder, single overhead cam with computer controlled, multi-point sequential port fuel injection.

The engine was removed intact from the automobile, and the accompanying wires and electronics were mounted on a panel to simplify operation. A special bellhousing with an adapter plate and a special clutch disc were made in order to adapt the motor to the SF 901 Dynamometer.

The motor was completely dismantled, inspected, and found to be in excellent condition. The pistons were removed and .075 inch deep relief pockets were machined in order to ensure that the inner valves of the vented valve units would have sufficient room to function without making contact with the pistons.

All relevant data were gathered while the engine was disassembled. (For example, the compression ratio of 7.8:1 was obtained using a cc burette.) The engine was reassembled and run, free load, on the engine stand. When all systems were determined to be functioning properly, the test engine was ready for the standard valve baseline testing.

A number of problems were encountered during the initial machining of the vented valves. The incorrect Rockwell hardness rating was provided for the stainless steel blanks used for the outer valves, which resulted in numerous delays. The inner titanium valves were machined easily.

Once the valve pieces were completed, the inner valves were lapped-in for proper sealing, and the springs and pins were adjusted and installed.

Engine Testing

Engine tests were conducted at Western Washington University, Bellingham, Washington, at Dr. Michael Seal's Vehicle Research Institute. Acro-Tech rented the dynamometer laboratory and mounted the engine in stock form of the SF 901 Dynamometer.

An exhaust probe from an exhaust gas analyzer was set up to track hydrocarbons and carbon monoxide emissions throughout the tests. Unfortunately, the NOx analyzer was malfunctioning at the time of the test and could not be repaired in time. Acro-Tech believes that these exhaust gases would not be problematic because exhaust gas temperatures dropped during the tests.

The exhaust emissions analyzer required a separate process to record the data manually as the dynamometer runs were conducted. This manual recording proved to be inefficient, and therefore the emissions data are not a thorough and precise as the dynamometer data. However, definite trends were revealed, and other factors must be analyzed in the context of real-life situations to determine the potential gain or loss.

Tests were conducted with a dynamometer, an instrument designed to analyze an engine's ability to resist precisely measured loads and calculate horsepower and torque. Today's dynamometers are computerized and can extract and analyze a great deal of data on such parameters as fuel efficiency, volumetric efficiency, and thermodynamics.

The Mitsubishi 2.0 liter, 4 cylinder ran well during the tests. Dynamometer runs over 6,000 RPMs were made without any problems. Once the data had stabilized over several dynamometer runs, Acro-Tech collected all relevant data from the stock engine.

The standard valves were then removed and the vented valves installed. Several special reamers enlarged the valve guides from 8 millimeters to 9 millimeters to accommodate the stem of the vented valves. The cc burette was again used to measure the volume of the combustion chamber, and the engine was reassembled.

The vented valve engine first was run slowly below 2,500 RPMs and was gradually increased to beyond 6,000 RPMs.

EVALUATION PARAMETERS

All data gathered on both engine configurations were corrected to Society of Automotive Engineers (SAE) standards, which mathematically corrects the ambient atmospheric conditions observed at the time of the test to a standard of 100 kilopascals absolute (29.38 inches mercury) inlet air pressure and 25 degrees centigrade (77 degrees fahrenheit) inlet air temperature. Dr. Michael Seal, Director of the Vehicle Research Institute, witnessed and certified the data, which include comparisons of horsepower, torque, brake special fuel consumption (BSFC), brake specific air consumption (BSAC), and air to fuel ratios.

ASSESSMENT SUMMARY

An analysis of various factors revealed that Acro-Tech had not achieved an optimum assessment of the technology's potential. After solving some minor timing problems, the modified vented valve engine started and ran smoothly with no noticeable increase in noise or vibration. An objective analysis also revealed an improved throttle response.

DEMONSTRATION COST

The total cost of the demonstration was \$48,042.72, with \$25,000 being covered by EPA's Pollution Prevention By and For Small Business program, and the balance of \$23,042.72 shared by Acro-Tech.

RESULTS AND DISCUSSION

TEST RESULTS

Project results indicate that vented valves do work and have a great deal of potential.

All critical data were tabulated, sorted, and organized by the dynamometer computer at any RPM point chosen by the dynamometer operator. This results in raw data from various RPM points. In order to plot evenly spaced points (e.g., 500, 1,000 1,500 RPMs), the data need to be interpolated using a mathematical formula:

$$Y2 = Y1 + (((Y3 - Y1) \div (X3 - X1))(X2 - X1))$$

where:

- X2 equals the desired evenly spaced data or RPM point
- Y2 equals the unknown value for that point
- X3, X1, Y3, and Y1 represent raw data points from the dynamometer

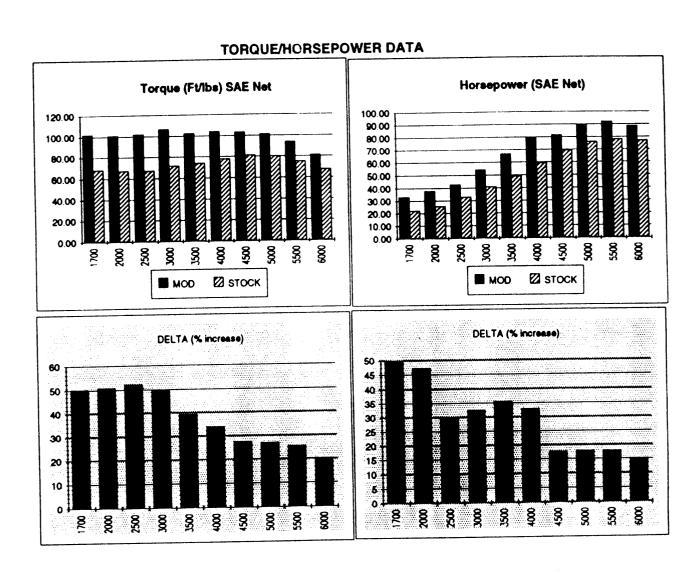
This yields credible data at evenly spaced data points that allows for graphing and making easy comparisons to other data within the project, and to data from other projects.

Torque and horsepower data for the stock engine and the vented valve engine are shown in Table 1 and Figure 4.

TABLE 1. Normalized Torque and Horsepower Date for Modified (Vented Valve) and Stock Engines

	Normalized Torque Data (Ft/Lbs) (SAE Net)			Normalized Horsepower Data (SAE Net)		
RPM	Vented	Stock	Delta %	Vented	Stock	Delta %
1,700	101.90	67.92	50.01619	33.00	22.01	49.89231
2,000	100.90	66.92	50.76715	37.50	25.48	47.16608
2,500	101.85	67.00	52.01624	42.50	32.84	29.41703
3,000	106.50	71.36	49.237	54.19	40.92	32.41595
3,500	102.42	73.66	39.03991	66.49	49.08	35.47362
4,000	103.98	77.74	33.75204	78.94	59.42	32.84486
4,500	103.15	81.00	27.35045	81.55	69.39	17.52843
5,000	101.47	79.96	26.89902	89.69	76.10	17.85621
5,500	93.83	74.92	25.24843	91.97	78.05	17.84094
6,000	80.95	67.37	20.16507	88.52	76.92	15.0893

Figure 4. Torque and Horsepower Data



A comparison of air/fuel consumption for the stock engine and the vented valve engine is shown in Table 2 and Figure 5.

TABLE 2. Normalized Air/Fuel Consumption Data for the Modified (Vented Valve) and Stock Engines

	Normalized BSFC Data (Lbs/Hp.Hr)			Normalized BSAC Data (Lbs/Hp.Hr)		
RPM	Vented	Stock	Delta %	Vented	Stock	Delta %
2,000	0.61	0.64	-5.98593	14.09	8.16	-42.1091
2,500	0.53	0.67	-20.4018	14.10	7.73	-45.1744
3,000	0.56	0.67	-15.8277	13.45	8.08	-39.9009
3,500	0.62	0.63	-1.14517	12.57	7.27	-42.1411
4,000	0.65	0.62	5.668204	10.34	6.91	-33.1594
4,500	0.67	0.70	-4.27512	10.17	6.87	-32.4564
5,000	0.69	0.74	-7.44707	9.54	7.19	-24.6909
5,500	0.72	0.76	-5.80395	9.59	7.86	-18.041
6,000	0.80	0.86	-6.43653	9.97	8.50	-14.67

BSFC: Brake Specific Fuel Consumption BSAC: Brake Specific Air Consumption

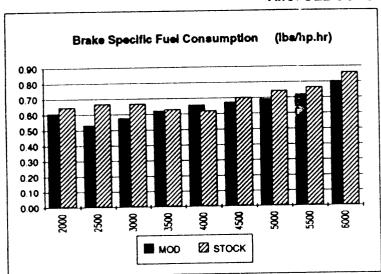
A comparison of hydrocarbon and carbon monoxide emissions data for the stock engine and the vented valve engine is shown in Table 3 and Figure 6.

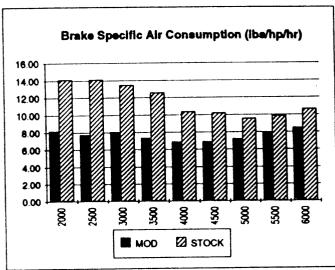
TABLE 3. Normalized Hydrocarbon and Carbon Monoxide Emissions Data for Modified (Vented Valve) and Stock Engines

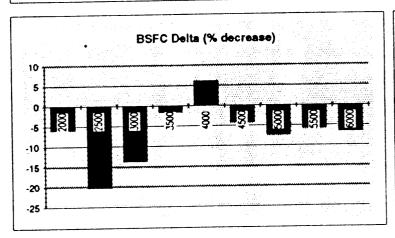
	Normalized Hydrocarbon Data (ppm)			Normalized Carbon Monoxide Data (%)		
RPM	Vented	Stock	Delta %	Vented	Stock	Delta %
2,000	265.48	202.27	31.24666	3.00	4.45	-32.5843
2,500	186.11	216.48	-14.0274	3.00	4.59	-34.627
3,000	193.22	229.59	-15.8401	3.00	4.72	-36.4407
3,500	190.87	241.06	-20.8189	3.20	4.85	-33.94
4,000	177.44	256.96	-30.9456	3.45	5.00	-30.9118
4,500	197.98	288.61	-31.4025	3.96	5.00	-20.809
5,000	200.00	316.67	-36.8421	4.67	5.00	-6.56371
5,500	200.00				4.82	
6,000	200.00				4.61	

Figure 5. Air/Fuel Consumption

AIR/FUEL CONSUMPTION







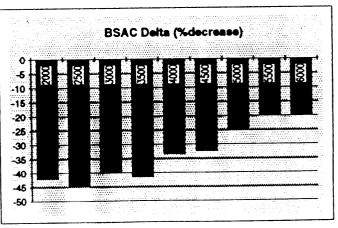
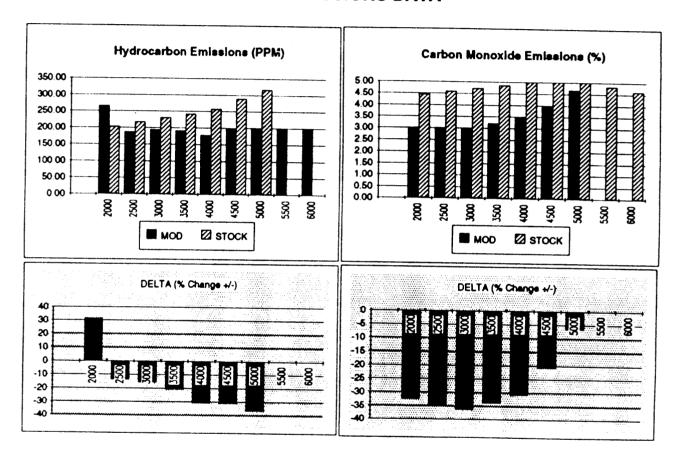


Figure 6. Emissions Data

EMISSIONS DATA



Reviewing this data illustrates the profound effect vented valves had on the test engine. Special attention should be given to the torque data, which indicates that an element of variable lift and timing was present, as a very flat torque curve was created (the flattest observed by Dr. Seal and Mr. Huff). The implications of this observation suggest the possibility of using lighter transmissions with fewer gears, giving vehicle designers another tool to increase efficiency.

The brake specific fuel consumption and brake specific air consumption are measured in pounds per horsepower hour, and are a direct gauge of the efficiency of the engine, indicating how much power the engine is producing relative to the amount of air and fuel consumed.

The emissions data are pre-catalyst; they represent the actual emissions measured without a catalytic converter or air pump applied to the exhaust system. The emissions were measured under fully loaded dynamometer testing.

FACTORS IMPACTING PERFORMANCE

One concern of using a late model engine with computer controlled ignition and fuel delivery systems was that if this technology exceeded the parameters programmed into the data computer, the performance results could be skewed. This apparently happened with the test engine. The dynamometer's computer indicated that the standard valve engine ran with better air/fuel ratios than the vented valve engine. This was caused by the tremendous increase in the vented valve's flow dimension and turbulence that "confused" the computer and/or sensors into thinking that the engine was running too lean (i.e., there was too much air to the fuel, or the air to fuel ratio was too high). The computer then increased the fuel flow, which actually resulted in the engine running far to rich (i.e., the ratio of air to fuel was too low).

Air/Fuel Ratio Comparison

The air/fuel (A/F) ratio comparison for the stock engine and the vented valve engine is shown in Table 4.

TABLE 4. Air/Fuel Ration Comparison for the Stock and Vented Valve Engines

Stock A/F	Vented A/F	Delta % Increase
16.7:1	12.3:1	357
16.0:1	12.5:1	28
15.4:1	12.8:1	20
14.7:1	13.7:1	072
13.4:1	11.0:1	21
13.1:1	10.4:1	25
11.9:1	10.5:1	13
12.2:1	10.5:1	16
	16.7:1 16.0:1 15.4:1 14.7:1 13.4:1 13.1:1 11.9:1	16.7:1 12.3:1 16.0:1 12.5:1 15.4:1 12.8:1 14.7:1 13.7:1 13.4:1 11.0:1 13.1:1 10.4:1 11.9:1 10.5:1

The modified vented valve engine ran at a distinct disadvantage to the standard valve configuration. The vented valve engine, with its increased velocity and turbulence, could have run leaner than the standard valve engine. Yet it outperformed its counterpart while running too rich. All data, especially concerning fuel efficiency and emissions, would have improved significantly had Acro-Tech been able to adjust the air/fuel ratio to optimum levels.

FAILURE ANALYSIS

Upon completion of all tests, the top of the engine was dismantled and the vented valves removed to inspect their condition following the testing. The vented valves were in perfect condition, with no abnormal wear, cracks, or blemishes. However, one of the inner valves was stuck fully closed, apparently caused by a piece of dislodged machining debris getting caught between the inner valve stem and guide. Acro-Tech cannot determine whether the valve was stuck throughout the test.

COST/BENEFIT ANALYSIS

Based on the data gathered, Acro-Tech believes that a 20 percent fuel savings could be realized with the vented valve engine over existing designs. Using 1989 U.S. new car sales, the approximate fuel savings can be calculated using a 1 percent, 5 percent, and 10 percent vented valve implementation. Reductions in liquid fuel consumption (cost savings to the consumer) are calculated to be:

- 1 percent = 11,840,922 gallons saved
- 5 percent = 59,204,610 gallons saved
- 10 percent = 118,409,220 gallons saved

For the manufacturer, vented valves offer the possibility of less mechanical complexity in order to obtain desired fuel efficiency and emissions standards, as well as the potential of requiring less exhaust catalyst and perhaps smaller, less expensive transmissions.

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Pollution prevention is largely associated with fuel savings. If two automobiles of equal weight and size burn fuel at totally different rates, it is likely that the auto burning the most fuel also emitted the most pollution.

If vented valves lead to the use of smaller engines and transmissions, this simplified manufacture could reduce pollution produced in the manufacturing process.

Currently, vented valve development is in the third stage of development and is nearing production of an aftermarket product. This will lead to extensive failure analysis and durability testing that could lead to an in-market production vehicle product.

Industry resistance to new product design from outside sources may prove to be a barrier to the acceptance of vented valves. Further testing and refinement to generate replicable data, as well as its success in aftermarket sales, can promote the adoption of this technology.

POLLUTION PREVENTION THROUGH USE OF A FORMALDEHYDE-FREE BIOLOGICAL PRESERVATIVE

by

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ABSTRACT

Formaldehyde, a major component of most biological preservatives, is a toxic air pollutant. 67 billion pounds of this substance are produced in the U.S. annually. Earth Safe Industries' product, NoToX, is a non-toxic, biological preservative intended to replace formaldehyde in a variety of applications. This project demonstrated the effectiveness of NoToX in eliminating air pollution (associated with the venting of formaldehyde) and acting as an effective, long-term preservative and fixative.

INTRODUCTION

PROJECT DESCRIPTION

Earth Safe Industries' objective for this project was to demonstrate that NoToX Biological Preservative and NoToX Histological Fixative could perform preservation and fixation tasks equivalently to formaldehyde in the biological and medical sciences. These tasks include anatomical embalming (needed for the study of gross anatomy), fixation and preservation of whole organs (used in research and autopsy investigation), and fixation and preservation of large tissue blocks and thin tissue sections (used to determine the absence or presence of disease states from surgical patients). Since NoToX Biological Preservative and NoToX Histological Fixative are made from non-toxic and non-carcinogenic constituents, pollution can be eliminated from the source. Approximately 5 million gallons of toxic waste is generated from formaldehyde use in the biological sciences annually; simply replacing NoToX where formaldehyde has traditionally been used eliminates this pollution. Earth Safe Industries' extensive testing of NoToX to meet the criteria of these applications found that NoToX is capable of replacing formaldehyde in all these protocols, and that researchers and health professionals can rely on NoToX to perform protocols equal to or better than formaldehyde. The data obtained from this study were instrumental in convincing formaldehyde users that performance would not be sacrificed for safety. NoToX Biological Preservative and NoToX Histological Fixative have made their way into the marketplace throughout the U.S., Canada, Europe, Mexico, and South America.

Product Outline

NoToX Biological Preservative is an environmentally safe anatomical embalming fluid developed specifically to meet the stringent requirements of anatomical preservation, both human and veterinary. Excellent long term anatomical preservation is achieved, and importantly, chemicals that are carcinogenic, toxic, and corrosive

(such as formaldehyde, phenol, and glutaraldehyde) have been completely eliminated. NoToX is formulated as an aqueous alcoholic solution consisting of protein stabilizers and modifiers, humectants, and potent antiseptic and antifungal agents. These constituents are regarded by regulatory agencies as environmentally and occupationally safe. With the absence of toxic and carcinogenic substances in the biological preservative, laboratory personnel can work safely with NoToX for unlimited periods of time. There is no irritation to the eyes and respiratory tract when personnel work with NoToX, nor is there the offensive, heavy, acrid odor commonly associated with formaldehyde and phenol. On the contrary, NoToX has a mild aroma and is a pleasant substance with which to work.

NoToX Histological Fixative has the long term preservation features of the biological preservative, but has been modified to meet the needs of histological tissue processing. Specimens prepared for histological evaluation are smaller and require less humectants, bactericides, and fungicides. The product does not sacrifice safety and performance, and has been optimized to meet this application traditionally delegated to a 10 percent neutral buffered formalin.

NoToX Histological Fixative features a unique chemical system that stabilizes a bis carbonyl compound and activates it towards a crosslinking reaction with protein. The NoToX crosslinker is selective and does not attack antigenic sites or DNA. Consequently superior results are obtained with immunohistochemical stains and *in situ* hybridization techniques. Although NoToX Histological Fixative can replace formaldehyde completely, NoToX Histological Fixative is interchangeable with formalinfixed specimens. Formaldehyde-fixed specimens arriving at a laboratory can be placed in a tissue processor containing NoToX and over-crosslinking will not occur. Thus, laboratories obtaining specimens from sources outside their facility need not disqualify their laboratory from using NoToX. Excellent results can be achieved when a specimen has initially been fixed in formaldehyde and then processed in NoToX Histological Fixative.

Unique Product Features

The elements of a common formula that produce superior tissue fixation unite the anatomical and histological applications. Key to the efficacy of NoToX Biological Preservative and NoToX Histological Fixative is a unique bis carbonyl compound that has the ability to crosslink protein in a manner similar to formaldehyde. The bis carbonyl compound is dissolved in an aqueous, alcoholic solvent and is fortified with high potency, non-toxic, antimicrobial agents that prevent the proliferation of bacterial. fungal, and viral pathogens. Alcohol not only functions as a supplementary protein denaturant, but also has the solvency power to dissolve certain lipophilic, germicidal compounds at much higher levels than could be attained in a purely aqueous medium. A polyhydridic alcohol, such as glycerin or propylene glycol, is included in the formulas as a humectant. The humectant is important in the anatomical application, as it retards the dehydration of unsubmerged or exposed tissue. In the histological application, unsubmerged tissue is never a problem. Since the polyhydridic alcohol has little, if any overall impact on the degree of protein denaturation, it can be omitted from the histological application with almost no loss of function. The polyhydridic alcohol has not been omitted from the histological formula, but has been reduced in concentration.

APPLICATION

Products Replaced

Four products emerged as a result of Earth Safe's studies. These are NoToX Biological Preservative and NoToX Histological Fixative, both of which replace formaldehyde and other toxic components used for preservation and fixation in the medical and biological sciences. Accessory products that are used in conjunction with anatomical preservation were also developed to replace current toxic counterparts. As the investigation was conducted, embalmers stated that in addition to the use of formaldehyde, either phenol or paraformaldehyde crystals were used to retard fungus formation on cadavers during storage. Some institutions had refrigeration facilities and wrapped cadavers in plastic bags that were stored in cold rooms. Paraformaldehyde crystals were sprinkled into the bags to inhibit fungus formation on the specimens. To replace these crystals and eliminate this source of pollution, an environmentally safe, non-toxic, non-carcinogenic, antifungal compound was developed with the trade name, VaporSafe. TankGuard was developed for those institutions using tanks for cadaver storage. These tanks, ranging from 30 gallons to 5,000 gallons, hold cadavers until needed. Phenol is added to the tanks to retard the formation of fungus. The environmentally safe TankGuard replaces the toxic phenol in this application. The development of these four products eliminated toxins off-gassed by formaldehyde, phenol, and glutaraldehyde.

NoToX Biological Preservative also proved to be an excellent replacement for formaldehyde in preserving dissection specimens (e.g., frogs, fish, cats, and rats) used in many biology classrooms. A major supplier of preserved animals successfully tested NoToX Biological Preservative as a replacement for its formaldehyde/glutaraldehyde fluid. Because of a downturn in the preserved animal market, the company opted not to adopt NoToX as its preservation fluid. Several schools of veterinary medicine (including Tufts University and the University of Montreal Schools of Veterinary Medicine) have adopted NoToX Biological Preservative to embalm cows, horses, goats, dogs, and cats for anatomical study.

NoToX Histological Fixative replaces formaldehyde in fixing and preserving biopsy specimens excised from patients or whole organs derived from autopsy examination, and formaldehyde as used in tissue processors in hospital and private medical laboratories. In these applications, approximately five million gallons of toxic formaldehyde waste can be eliminated annually.

Wastes Prevented

Formaldehyde, phenol, glutaraldehyde, and toxic substances off-gassed by paraformaldehyde crystals are eliminated at the source with the use of NoToX Biological Preservative, NoToX Histological Fixative, VaporSafe, and TankGuard.

NoToX Biological Preservative has the capability of eliminating 100,000 gallons of formaldehyde and phenol waste generated annually by medical, dental, and veterinary schools from anatomical embalming. Another source of formaldehyde pollution is dialysis machines. Approximately 100,000 gallons of formaldehyde are used to disinfect components from dialysis machines annually. Since NoToX

Biological Preservative has potent disinfecting capabilities, it can replace formaldehyde in this application and eliminate the waste formaldehyde generated.

Approximately 5 million gallons of formaldehyde waste is generated annually in the U.S. from the use of tissue processors used to prepare tissue samples for microscopic evaluation. Hospitals and private medical laboratories can save as much as \$140 million annually on waste disposal costs simply by switching to NoToX Histological Fixative and eliminating the generation of the formaldehyde waste.

Cross Segment Uses

As a formaldehyde substitute, potential cross industry uses for NoToX include:

- **Veterinary, medical, and dental schools** that embalm human and animal cadavers for anatomical study in the training of health professionals. NoToX Biological Preservative is presently being used for this application.
- Medical examiners' offices perform autopsies and preserve whole organs in large quantities of formaldehyde. NoToX Biological Preservative can preserve whole organs by passive immersion better than formaldehyde, as NoToX's selective crosslinking mechanism permits infiltration into the core of the organ. Formaldehyde tends to fix specimens on the outer parts of the organ since its crosslinking mechanism is random, which means that the core of the organ --particularly whole brains -- is not always fixed.
- Preservation of animals and specimens for dissection may be achieved with NoToX Biological Preservative. Testing was done with NASCO, a major worldwide supplier of preserved specimens to schools, universities, and research institutions that validated the utility of NoToX Biological Preservative in this application.
- NoToX Biological Preservative may be applied as an embalming fluid for the commercial funeral industry. NoToX's long term preservation capabilities have already been proven in medical and veterinary schools and may meet the needs of the commercial funeral industry.
- In the dialysis industry, NoToX Biological Preservative may be tested to replace formaldehyde to disinfect dialysis components.
- Hospitals, private medical laboratories, and research institutions may be
 able to eliminate formaldehyde in selected applications for tissue processing.
 NoToX Histological Fixative performs on a par with formaldehyde that is
 diagnostically equal as shown in the histological portion of the project at Mount
 Sinai Medical Center.

PROCEDURE

DEMONSTRATION

The NoToX project was divided into three categories: **Anatomical**, **Histological**, and **Microbiological**.

Anatomical Demonstration

The anatomical phase of the project was conducted under the direction of Mr. Roger Faison, Principal Investigator and Supervisor of the Gross Anatomy Department at the University of Medicine and Dentistry of New Jersey in Newark. Cadavers were embalmed with NoToX Biological Preservative and allowed to incubate under refrigerated storage conditions for varying periods of time: 1, 3, 6, 9, and 12 months. The objectives were to assess preservation and to determine if minimum and maximum time requirements existed for achieving good preservation. At the end of the incubation period, effectiveness of preservation was demonstrated by having students dissect the cadavers in their usual manner. Examination of internal and external regions of the cadavers would indicate effectiveness of the NoToX. In some cases, cadavers were stored at room temperature to determine whether NoToX would be effective in institutions without refrigeration facilities.

Histological Demonstration

Dr. Pam Unger of Mount Sinai Medical Center's Department of Pathology in New York City served as Principal Investigator for the histology section. Fifty one surgical specimens were fixed in neutral buffered formaldehyde and NoToX Histological Fixative and processed using standard histological protocols that were established for their laboratory. For all surgical cases, protocols for formaldehyde-fixed tissue and NoToX fixed tissue were identical. After slides had been prepared, the tissue samples were evaluated microscopically and compared.

Microbiological Demonstration

As a subsection of the Anatomical investigation, blood was drawn from cadavers before and after embalming. The blood was cultured relative to a representative panel of bacterial and fungal organisms. Colonies were counted in the pre- and post-embalmed samples to determine the reduction in inhibition of the organisms by NoToX. In another part of this study, which was conducted as part of the optimization procedure, microbial inhibition of NoToX was compared to formaldehyde and phenol by means of the Minimum Inhibitory Concentration and Kirby Bauer disc inhibition methods.

EVALUATION PARAMETERS

Environmental

While formaldehyde, phenol, or other toxic substances are not used in the NoToX formulations, Earth Safe elected to determine that no off-gassing of formaldehyde occurred through decomposition, or any other means. Unlike formaldehyde, there is no possible degradative mechanism by which phenol could

evolve. Levels were assessed merely as a point of validation, since phenol is always used together with formaldehyde in conventional anatomical embalming compositions. In the Anatomical Demonstration, where large amounts of the fluid were used for embalming cadavers, airborne concentrations of formaldehyde, phenol, and ethanol were measured with conventional air sampling equipment. Ethanol was sampled because NoToX is formulated with ethanol, and there is a remote chance that under certain conditions, levels may become elevated. In the Histological Demonstration, where relatively small amounts of the NoToX Histological Fixative are handled by technicians, air sampling is not necessarily a preferred method for monitoring levels of formaldehyde. The accepted method for monitoring personnel who are exposed to formaldehyde, in this case, is by requiring them to wear passive dosimeter badges. This was done for the personnel who were working with NoToX in the histology laboratory even though the likelihood of observing an elevated value was remote. Since ethanol levels in the histology laboratory are never considered a health threat, they are not measured. Therefore, they were not measured for NoToX either. Since phenol is not used in the histology laboratory, levels were not determined. Any level of phenol, regardless of how low, could be detected without instrumentation. although quantification would not be possible. None of the technicians or other handlers of NoToX reported the detection of phenol. Preclusion of the determination of phenol concentrations in this case, is justifiable on the grounds that the action level for phenol is 2.5 ppm, but the odor threshold is 0.04 ppm. Phenol can be detected by smell well before it rises to a danger level.

Anatomical

Preservation efficacy of the embalmed cadavers was judged by the following criteria: internal and external color of the cadavers; general appearance; firmness of internal and external regions, including the abdominal area and internal organs; ease of separating and identifying tissue; ability to identify fascial planes; and ability to maintain the integrity of muscle tissue.

Histological

The most important criteria for determining the effectiveness of NoToX Histological Fixative was that it provide diagnostic results that were equivalent to formaldehyde. Results that were too divergent, even if superior, might not be acceptable. Parameters used for judging NoToX Histological Fixative's utility were histologic criteria -- ease of processing and cutting specimens -- and microscopic criteria -- stain intensity for both conventional and immunohistochemical stains, cellular architecture, nuclear detail, and contrast.

Microbiological

The reduction in organism colonies after embalming was the key parameter in the microbiological study. With respect to the two other microbiological studies previously mentioned, the objective was to compare the potency of NoToX Biological Preservative to formaldehyde and phenol-based preservatives.

ASSESSMENT SUMMARY

Environmental

As part of the Anatomical Demonstration, assessment of airborne levels of formaldehyde, phenol, and ethanol were determined with conventional air sampling equipment from "Alltech." Background levels of formaldehyde had to be determined because cadavers that had been previously embalmed with formaldehyde were being stored in the same general area of the anatomy laboratory as were the NoToXembalmed cadavers. In general, the NoToX cadavers were kept as far from the formaldehyde/phenol cadavers as possible, but it was impossible to prevent some commingling of the vapors. Sample air measurements were made by keeping the probe about one foot from the cadaver at a point approximately 1.5 feet above the platform of the dissection table. Background measurements were made in the laboratories on a day when no embalming or dissection activity of formaldehyde/phenol cadavers was being conducted. For background measurements, sampling probes were positioned with respect to cadavers as they would be when actual embalming or dissection activity was taking place. Since the entire laboratory area was permeated with the odor of phenol, background measurements had to be made with the same consideration as was explained above for formaldehyde.

Ethanol is not used in the UMDNJ laboratories. Their normal procedures call for large quantities of isopropyl alcohol. Ethanol is non-toxic with a TLV of 1,000 ppm, but isopropyl alcohol is a toxic substance with at TLV of 400 ppm. Earth Safe's concern was with the specificity of the sampling equipment. Earth Safe felt, that despite the manufacturer's claims, the chemical similarity of ethanol to isopropyl alcohol would tend to give false positive values for airborne ethanol. Background and sample levels of ethanol turned out to be so low and so far from the TLV value, that concerns over specificity were unfounded.

Measurements of the above substances were made in the laboratory used for embalming as well as that used for dissection (student gross anatomy lab). Levels of formaldehyde were always at or below background levels (0.6 ppm) in all critical areas where NoToX was utilized. The permissible exposure limit (PEL) for formaldehyde is 0.75 ppm. Airborne concentrations of phenol were below background levels (less than 1 ppm) in all critical laboratory areas where NoToX was utilized. The PEL for phenol is 5 ppm. Background and sample values for ethanol were about 0.3 and 13 ppm respectively, a small fraction of the 1,000 ppm TLV value. Typical data that were collected for air sampling in different areas are shown in Table 1 in the Tabulation of Data section.

Formaldehyde dosimeter badges from "Alltech" were worn by technicians who were working with formaldehyde during the Histological Demonstration. Values of formaldehyde were always less than the background value, which was 0.2 ppm.

Anatomical

No difference in preservation ability was noted for specimens stored at the 1, 3, 6, 9, or 12 month intervals. Fixation occurred rapidly so that good preservation was observed before 1 month, and that long term preservation was of sufficient quality so

that a well produced specimen was still retained after 12 months of storage under refrigeration conditions. Likewise, cadavers stored at room temperature were of preservation quality equal to those stored in the cold. The overall appearance of the preserved cadavers was quite good. External colors had a pale yellow, natural appearance in contrast to the grey-brown color of cadavers embalmed with formaldehyde/phenol solutions. Extremities were well-preserved, and mold and fungal growth on cadavers was not observed. Textures were firm but not dehydrated or mummified as would be the case if over-crosslinking had occurred. In particular, the abdominal region felt firm on all specimens. Upon dissection, the integrity of muscle tissue appeared well-maintained, tissues were easy to separate and identify, and fascial planes were readily identifiable. Internal organs, such as the heart and intestines, were firm.

Histological

The significant conclusion drawn from this study was that NoToX Histological Fixative is diagnostically equal to formalin. Tissue fixed in NoToX Histological Fixative showed no significant differences either in the processing of tissue or in the cellular morphology as judged by the criteria above. Optimal results were obtained without vacuum in the fixation chamber. NoToX Histological Fixative showed weaker staining of cytoplasmic granules, but otherwise, cytoplasmic staining was comparable. Eosin and immunohistochemical stains showed greater stain intensity, but diagnostically this is not perceived as being a problem, since in the case of eosin, the greater stain intensity makes for greater contrast. NoToX Histological Fixative, in contrast to formaldehyde, does not destroy antigenic sites, which allows for greater stain intensity in immunohistochemical stains. Since there is a greater amount of antigen available for reaction with antibody, the immunohistochemical stain becomes too intense and high background stain results. Rather than changing the fixative formulation, a better approach would be to instruct the user to titer the antibody concentration for optimal contrast. By using 25 percent less of the amount of antibody normally used for formaldehyde-fixed tissues, superior immunohistochemical stains can be achieved. This practice saves money for the user by reducing the amount of antibody needed for immunohistochemical staining.

Microbiological

Fluid withdrawn from a cadaver that had been embalmed for one month showed zero population colonies for a panel of bacterial and fungal organisms including E. coli, P. aeruginosa, S. aureus, A. niger, and C. albicans. A microbiological study conducted by Earth Safe Industries was designed to generate comparative minimum inhibitory concentration (MIC) values for NoToX Biological Preservative, formaldehyde, and phenol. The results of this testing revealed that NoToX had antiseptic potency equal to formaldehyde and 5 times greater than phenol. In a comparative Kirby Bauer disc inhibition study also conducted by Earth Safe, the results found that NoToX had fungicidal activity at least twice that of formaldehyde and 5 times that of phenol.

COST/PAYBACK OF DEMONSTRATION

The final cost of the demonstration was \$35,653, with \$25,000 provided by EPA through the Pollution Prevention By and For Small Business Grant Program.

RESULTS AND DISCUSSION

PERFORMANCE RESULTS

Anatomical Performance

NoToX Biological Preservative was used effectively as an anatomical perfusion fluid. preserving cadavers as well as formaldehyde and phenol embalming solutions. Optimal results were obtained when the fluid was perfused through the carotid artery at 20 pounds of pressure using 8 to 10 gallons of fluid. Supplemental injections to the abdominal region enhanced fixation of the viscera. Cadavers embalmed with NoToX were firm, yet pliable and had a lifelike texture that made them well-suited for medical school, anatomical, and surgical dissection. Some surgeons preferred NoToX embalmed specimens for practicing surgical techniques. A member of the Arthroscopy Association of North America evaluated knee joint specimens preserved in NoToX and indicated that these were viable alternatives to fresh frozen specimens used in teaching laser surgery techniques to orthopedic surgeons. In addition to demonstrating long term preservation, NoToX Biological Preservative also showed its ability to inhibit microbial proliferation as an embalming solution. Since the two fundamental criteria of anatomical embalming were met, it was concluded that NoToX Biological Preservative is an effective substitute for formaldehyde embalming fluids. Air levels were measured around specimens embalmed in NoToX and there was no off-gassing of toxic substances. Students found NoToX embalmed specimens did not exhibit unpleasant odors.

Histological Performance

Tissue fixed in NoToX Histological Fixative was diagnostically equal to tissue fixed in formaldehyde. There are some minor differences between NoToX and formaldehyde fixed tissue, but these do not affect its diagnostic utility. NoToX gives a light tannish appearance to the tissue that some laboratorians may misconstrue as unfixed specimens. However, upon touching and evaluating the tissue microscopically the specimens are indeed firm and preserved.

Red blood cells can be seen in NoToX-fixed tissue, but they appear differently than red blood cells that are seen in formaldehyde-fixed tissue. In NoToX, the red blood cells look like flat, two-dimensional, light orange, outlined discs. The change in appearance of the red blood cell does not affect cellular architecture, which is the crucial factor for medical diagnosis.

Specimens fixed in NoToX and specimens fixed in formaldehyde were compared side by side and judged on the criteria of cellular architecture, nuclear detail, cytoplasmic detail, and strength of contrast. The principal investigators at Mount Sinai Medical Center determined that the NoToX-fixed specimens were diagnostically equal to the formaldehyde-fixed specimens.

PRODUCT QUALITY VARIANCE

Product quality variance is not an issue. NoToX Biological Preservative and NoToX Histological Fixative come ready to use and can be made with a high degree of consistency from lot to lot. Any performance variables are due more to the

individual techniques of end users. If established protocols are followed, the end user will obtain effective results. For example, Earth Safe recommends that NoToX Biological Preservative be perfused through the carotid artery at 35 pounds of pressure and that 8 to 10 gallons of fluid be used for optimal preservation. NoToX Histological Fixative is recommended to be used without dilution to achieve optimal results. When used according to directions, there is no variation in fixation or preservation quality.

CONDITIONS THAT IMPACT PERFORMANCE

NOToX Biological Preservative and NoToX Histological Fixative usually can be used like formaldehyde. However, neither NoToX product can be diluted as is sometimes done with formaldehyde. Some minor changes in protocols enhance performance.

Embalming Procedures

Adequate fluid should be used for perfusing anatomical specimens. Earth Safe recommends eight to ten gallons per cadaver. The choice of perfusion artery sometimes affects results. The most consistent results have been obtained when the cadaver is perfused through the carotid artery. Supplemental injections to the abdominal region are helpful in ensuring that viscera reach a firm texture. Alternately, the viscera can be placed in a pail containing NoToX Biological Preservative after dissection has begun; viscera will continue to firm up within 24 hours. A 30-day curing period is recommended before dissection is begun.

NoToX Biological Preservative works well with specimens that are stored at room temperature and under refrigerated and freezing conditions. In geographic locations with high humidity, VaporSafe or TankGuard may be used in conjunction with NoToX Biological Preservative to fortify the inhibition of fungus formation on the cadavers. This also would be true when formaldehyde is used to embalm cadavers.

Histological Tissue Processing

NoToX Histological Fixative is compatible with automated histological tissue processors and has been approved for use by Miles on their VIP series. Although Miles is the largest manufacturer of tissue processors, other brands can be found in hospitals and privately owned laboratories. Brand and age of the processor may affect the results. Simple adjustments can be made to the settings on the processor. Adjustments to the temperature setting, vacuum setting, or time of cycle setting may be required. Adjustments to the automatic settings on tissue processors using formaldehyde also are required at times to prevent over-fixation of specimens.

TABULATION OF DATA

Environmental Demonstration

Measurements of analytes that resulted from air sampling during the anatomical demonstration are shown in Table 1.

TABLE 1. Airborne Concentrations of analytes with NoToX Procedures

DATE	LOCATION	ACTIVITY	ANALYTE	AIR CONC. (PPM)
3-23-92	embalming lab	background check	formaldehyde	0.6
3-23-92	embalming lab	background check	phenoi	0.2
3-23-92	embalming lab	background check	ethanol	0.3
3-26-92	embalming lab	embalming	formaldehyde	0.3
3-26-92	embalming lab	embalming	phenol	0.2
3-26-92	embalming lab	embalming	ethanol	13
6-2-92	embalming lab	background check	formaldehyde	0.3
6-2-92	embalming lab	background check	phenol	0.8
6-2-92	embalming lab	background check	ethanol	0.6
6-9-92	embalming lab	embalming	formaldehyde	0.4
6-9-92	embalming lab	embalming	phenol	0.1
6-9-92	embalming lab	embalming	ethanol	14
7-6-92	gross lab	dissection	formaldehyde	0.2
7-6-92	gross lab	dissection	phenol	0.1
7-6-92	gross lab	dissection	ethanol	2

Histological Demonstration

Fifty one surgical specimens were processed identically in NoToX Histological Fixative or neutral buffered formalin (NBF). They were fixed for 24 hours, embedded in paraffin, then cut and stained with hematoxylin and eosin. Four features were assessed on histological examination: architecture, nuclear detail, cytoplasmic detail, and strength of contrast. Architecture, nuclear detail, and cytoplasmic detail were evaluated to determine disease states. Strength of contrast refers to staining compatibility for ease in distinguishing cellular components. Numerical values were assigned with 3+ being the highest value and 2+ the lowest. Drs. Unger and Hague indicated that the numerical values were subjective and that the two fixatives -- NBF and NoToX -- were very close, practically indistinguishable. On certain tissue types, NoToX produced superior results. These were on fatty and muscle tissue such as breast and uterine specimens. After completing the study, Drs. Unger and Hague

concluded, "Overall diagnostic quality of tissues was maintained in tissues fixed in NoToX. NoToX Histological Fixative can give pathologists the quality of sections they require, as well as a product that is occupationally and environmentally safe. All of the immunohistochemical studies performed revealed no differences in quality or quantity of staining of NoToX and formalin fixed tissues."

Microbiological Data

The objective of this study was to determine the antimicrobial effectiveness of NoToX Biological Preservative and NoToX Histological Fixative. Formaldehyde has antimicrobial properties as well as preservation properties. Phenol is commonly used in conjunction with formaldehyde to enhance the antimicrobial effectiveness of formaldehyde, particularly in the preservation of human and animal cadavers. To evaluate NoToX Biological Preservative and NoToX Histological Fixative's ability to inhibit pathogens, two studies were conducted: an MIC Study and a Kirby Bauer Disc Inhibition Study.

The purpose of the MIC was to show the lowest concentration at which a preservative would exhibit antimicrobial activity. (See Table 2.) In this study, NoToX, formaldehyde, and phenol were diluted until they lost antimicrobial activity (dilutions below 3 percent were not attempted). The lower numbers represent greater potency. For example, NoToX was effective against E. coli when diluted to a concentration of 3 percent of its normal concentration, whereas phenol could only be diluted to 25 percent of its normal concentration before it lost potency. In the case of A. niger, a fungus, NoToX and formaldehyde still had potency at 3 percent of their normal concentrations, whereas phenol was not even effective at the 100 percent level. Earth Safe's results from this study found that NoToX has antiseptic potency equal to formaldehyde and 5 times greater than that of phenol.

TABLE 2. MIC Values (%) for Bacteria and Fungi

Preservativ e	E. coli	B. meg	P. aerug	S. aureus	A. niger	R. stolonifer
NoToX	3	3	3	3	3	3
10% Formalin	3	3	3	3	3	3
5% Phenol	25	NI*	12	50	100	25
CaroSafe†	NI	NI	NI	NI	NI	NI

^{*} No inhibition

The Kirby Bauer Study was conducted to compare antifungal potency for various preservatives as shown in Table 3. In this study, paper discs were impregnated with the preservatives and placed in a nutrient broth that would grow the organism of interest. The numbers reported in Table 3 indicate diameters of zones in which a microorganism could not grow. With respect to A. niger, the inhibition zone for NoToX was 60 millimeters, that for formaldehyde was 32 millimeters, and that for

⁺ Preservative manufactured by Carolina Biological

phenol was 8 millimeters. This indicates that NoToX has superior potency against the fungal organism A. niger. For the organism R. stolinifer, NoToX showed the inhibition zone of 38 millimeters while formaldehyde and phenol showed 0 inhibition zones. The interpretation of the disc sizes indicates that NoToX has fungicidal activity at least twice that of formaldehyde and 5 times that of phenol.

TABLE 3. Size of Disc Inhibition Radii (mm) Relating to Kirby Bauer Anti-fungal Disc Inhibition Study

Preservative	A. niger	B. stolonifer
NoToX	60	38
10% Formalin	32	0
5% Phenol	8	0

COST/BENEFIT ANALYSIS

Formaldehyde has been in use for over 130 years and is a relatively inexpensive fixative for initial costs. However, in today's regulatory and environmental climate many indirect costs impact formaldehyde use. These include the costs of hazardous waste disposal, training and safety programs for employees exposed to formaldehyde vapors, administrative costs to meet OSHA and EPA standards, monitoring expenses, safety gear, installation and maintenance of engineering controls, and the liability of Medical Removal Protection for each employee exposed to formaldehyde. The potential liability also exists for OSHA fines when permissible air levels go above the 0.75 ppm standard. These fines have ranged from \$5,000 to over \$40,000 per incident.

All of these liabilities can be eliminated when NoToX is adopted in the laboratory. If the purchasing agent considers the indirect costs of formaldehyde use and compares them to the direct cost of the NoToX products, she will find that NoToX is a highly cost effective measure. Table 4 shows a sample waste disposal cost comparison of NoToX Histological Fixative and formaldehyde for a histological application.

TABLE 4. Waste Disposal Cost Comparison: Histological Application of NoToX Versus Formaldehyde

Product	Ave. Direct Cost (per gallon)	Ave. Disposal Costs (per gallon)*	Total Cost (per gallon)		
Formaldehyde	\$10	\$21	\$31		
NoToX H.F.	\$17	\$0	\$17		
Savings derived when NoToX is used\$14 per gallon					

*assuming: a hazardous waste disposal cost of \$1,300 per 55-gallon drum, based on a quote from S&W Waste, NJ; 50 gallons of a 55 gallon drum of formaldehyde would be wasted and disposed as hazardous

When these disposal costs are factored into the direct cost of formaldehyde (which averages about \$10 per gallon) the actual costs of using formaldehyde rises to \$31 per gallon or more. The average cost of NoToX is \$17 per gallon, which means there is an actual savings of \$14 per gallon.

Considering that there are 7,000 hospitals and private laboratories in the U.S. using as much as 5 million gallons of formaldehyde annually, the total savings that can be derived from using a formaldehyde replacement such as NoToX Histological Fixative could be significant.

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Incentives

The testing and research done on NoToX Biological Preservative and NoToX Histological Fixative during the project period resulted in a technology that was optimized for market acceptance. The regulatory climate surrounding formaldehyde may accelerate the acceptance of these products into the marketplace. (For example, OSHA lowered the permissible levels of formaldehyde exposure to 0.75 parts per million (ppm), further encouraging laboratories to at least evaluate NoToX Biological Preservative and NoToX Histological Fixative.) Finding a formaldehyde replacement that would perform *all* tasks traditionally delegated to formaldehyde has been a major problem to those who wanted to discontinue formaldehyde use. NoToX is filling that need in the marketplace and is being distributed worldwide and gaining acceptance.

NoToX Biological Preservative and NoToX Histological Fixative reached their final phase of development, and no further developmental work is planned. Some product improvements may be considered in the future, but the products are now performing to meet the needs and expectations of the marketplace.

NoToX Biological Preservative is targeted for several markets. These include anatomy laboratories at medical, dental, and veterinary schools; the funeral industry; companies dealing in artificial organ components; and companies dealing with dialysis supplies.

NoToX Histological Fixative is targeted for hospital and private medical laboratories as well as research institutions. The Histological Fixative also is being used as an environmentally safe fixative for parasitology and is being distributed by Scientific Device Lab, Chicago, Illinois, which caters to the parasitology and microbiology market.

Another event that enhanced the credibility of NoToX Biological Preservative and NoToX Histological Fixative was the R&D 100 award that designated NoToX as one of the 100 most technologically significant products of the year. NoToX Biological

Preservative and NoToX Histological Fixative are now being marketed worldwide. The study provided the opportunity to fine tune the formulas to ensure they would meet the needs of end users.

Barriers

There appears to be no technical shortcomings. Pricing has been somewhat of an issue since the customer is accustomed to paying a nominal price for formaldehyde. However, as the customer becomes educated about the true costs of formaldehyde use, the price of NoToX becomes less of a barrier.

The solution to overcoming the pricing barrier is to educate the consumer by meeting them at national and regional association meetings. Salespeople are making direct contact with these consumers, and telemarketing, advertising, and direct mailing efforts are used to highlight the cost effectiveness of NoToX. Another solution to the pricing barrier is manufacturing the product more cost effectively which will occur as the volume of demand increases.

SUBSTITUTION OF BIODEGRADABLE LOW TOXICITY NATURAL PRODUCTS FOR THE KILLING OF FIRE ANTS

by

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ABSTRACT

Environmental Pesticides Group found that a low toxicity, biodegradable, natural products (terpines isolated from citrus fruit peel) can be substituted for toxic chemicals for the effective killing of fire ants in agricultural settings or on residential lawns. The fire ant has invaded the southern United States causing great damage to farm animal production, agricultural product harvesting, native ant populations, and recreational lands. This project showed that citrus fruit terpene compounds are (1) as effective as commonly used insecticides in killing fire ants; (2) less expensive than commonly used toxic chemicals; and (3) rapidly biodegradable in the environment.

INTRODUCTION

PROJECT DESCRIPTION

The fire ant, *Solenopsis invicta*, a native of South America, was inadvertently introduced into the United States at the port of Mobile, Alabama in the 1930s. Since that time, it has spread throughout the southeastern and southcentral U.S. where it currently occupies over 250 million acres, spanning all or parts of 11 states and Puerto Rico. Its aggressive nature, potent sting, large colony size (upwards of 250,000 workers per nest), and feeding and mound building habits make this ant a particularly noxious pest. The problems caused by this species are numerous and include stinging people and their animals, harming wildlife, defacing lawns and parks, and interfering with farming operations. The problem is growing worse as the ant continues to expand its range northward and westward. Should the ant become established on the West Coast, it is expected to thrive throughout coastal California, Oregon, and Washington.

In Texas, about half the state -- 65 million acres -- is infested and under a United States Department of Agriculture (USDA) quarantine restricting the movement of many products and materials from these areas to uninfested areas. The Texas Department of Agriculture has estimated that the fire ant costs the state at least \$31 million per year in losses and damages. In the infested areas, farmers, commercial turf and plant producers, and home owners are engaged in a constant battle to protect their products and homes. In order to fight this battle they are using a wide spectrum of toxic chemicals on a routine basis. The Texas Department of Agriculture has published a list of over 100 products registered for fire ant control; all are toxic to humans and other mammals and pose some threat to the environment.

The most popular form of fire ant control remains the spraying of toxic chemicals by agricultural concerns, residential pesticide companies, and home owners. These chemicals and their by-products contribute significantly to residuals found in public wastewater treatment plants, public water supplies, and residential yards. Development of a non-toxic, cost effective method of fire ant control would greatly reduce pollution (through source reduction) caused by the thousands of small business agricultural sprayers, residential exterminators, and home owners in the fight against this noxious pest.

The Environmental Pesticides Group is perfecting a non-toxic, unique fire ant pesticide that is highly effective in controlling *S. invicta* and replaces the toxic substances currently in use. The product is a blend of two environmentally safe ingredients with water. The active ingredient, d-limonene, is a natural terpene product isolated from citrus fruit peel. D-limonene is blended with Mazclean, an emulsifying agent, and the resulting mixture is diluted with water to achieve a solution containing 3 to 5 percent d-limonene. This final formulation, called Dead Ant, has no known toxicity, flammability, or environmentally hazardous effects. Environmental Pesticides has found that Dead Ant can be as effective as commonly used toxic insecticides in killing fire ants in both the laboratory and a commercial field setting. In addition, the active ingredient, d-limonene, degrades rapidly, almost disappearing from the soil in four days.

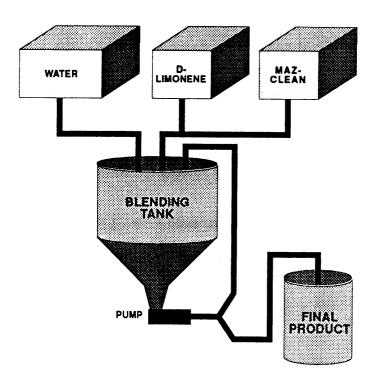
Unique Product Features

The unique properties of Dead Ant come from the unusual way in which it appears to affect the target pest. Although not scientifically documented, the observations of Environmental Pesticides Group suggest that d-limonene destroys the outer protective layer of the ant cuticle resulting in death. Dead Ant works on contact and permeates the soil quickly, rapidly killing the workers, larvae, and queens. The product's major advantage is that it is non-toxic and does not introduce harmful chemicals into sensitive areas (e.g., yards, playgrounds, workplaces, barns, and the like). D-limonene is listed by the United States Food and Drug Association (FDA) and Federal Emergency Management Association (FEMA) as GRAS (generally recognized as safe), permitting its use in cosmetics, and in the food industry as an additive for flavoring and coloring foods and beverages. The emulsifier, Mazclean EP, is not considered a hazardous chemical or a priority pollutant by the manufacturer's (PPG Industries) Material Safety Data Sheet.

Process Schematic

The product preparation is shown in Figure 1. First d-limonene is blended with the emulsifying agent, Mazclean. The resulting mixture is blended with water in a large blending tank. The final solution, containing 3 to 5 percent d-limonene, is then pumped into smaller containers.

Figure 1. Dead Ant Product Preparation



APPLICATION

Products Replaced

The Environmental Pesticides Group found that a class of low toxicity, biodegradable, natural products -- terpines isolated from citrus fruit peel -- can be may substitute for toxic chemicals such as diazanon, dursban, malathion, arsenic, bromide gas, and trichloroethane for effective control of fire ants. Use of natural terpenes for fire ant control is a novel concept; Environmental Pesticides Group was unable to find published records of the application or the effectiveness of d-limonene for killing fire ants. The retail cost of Dead Ant is expected to be comparable or less expensive than other commonly used toxic products.

Wastes Prevented

The major waste prevention afforded by Dead Ant is its lack of environmental contamination. The product will not contribute to residuals found in public wastewater plants, public water supplies, and residential yards because it is non-toxic and rapidly degrades in the environment. The precise cost of residual pollution due to the current fire ant control products is difficult to estimate. However, the reduced risks to human health and the environment is expected to be considerable if Dead Ant was to be used extensively in place of other currently available products.

Cross Segment Uses

In addition to the control of fire ants, Dead Ant has excellent potential for control of other pest ants such as the Pharaoh's ant, Monomorium pharaonis, and the carpenter ant, Camponotus pennsylvanicus. Other insect pests may also be controlled with the product, and Environmental Pesticides Group is planning future tests with d-limonene.

PROCEDURE

Environmental Pesticides' project consisted of three studies: (1) laboratory studies of fire ant mortality; (2) field studies of fire ant mortality; and (3) product degradation under field conditions.

DEMONSTRATION PROCEDURES

University of Texas Laboratory Studies of Dead Ant Efficacy

Laboratory tests were conducted by Dr. Edward Vargo at the University of Texas at Austin. Source colonies for this study originated from the Brackenridge Field Laboratory of the University of Texas at Austin and were collected on 23 April 1992. Tests were conducted 26 April-21 May 1992. All colonies were of the multiple queen (polygyne) form. Small standardized treatment colonies were constructed consisting of 10 grams of workers and brood (all immature stages of fire ants) -- approximately 5,000 workers, 5,000 worker pupae, and 5,000 larvae -- and 5 queens. The ants were housed in plastic trays (40 x 27 x 9 centimeters with Fluon®-coated sides to prevent escape), each containing a nest (a 14 centimeter diameter Petri dish half-filled with moist plaster). The ants were able to move around in the trays. The test colonies were kept at room temperature, approximately 75°F. There were 5 replicate test colonies per treatment. Each treated colony received approximately 0.5 fluid ounces of product delivered as a fine mist with a hand held spray bottle. This method of application was enough to ensure that almost every exposed ant was coated with material.

Two methods of application were tested, simulating mound drench and surface spraying. Simulation of mound drench was performed by treating the colonies with the nest cover removed so that all ants, including brood and queens, were exposed and sprayed directly with the solution. Simulation of surface spray of the mound was achieved by treating the colonies with the nest covers on, thereby shielding those ants -- brood, queens, and approximately 80 percent of the workers -- that would normally remain below ground, unexposed to direct spray. Four concentrations of d-limonene solution were tested:

- 1.5 percent: 1.5 percent d-limonene, 0.75 percent Mazclean emulsifier
- 3.0 percent: 3.0 percent d-limonene, 1.5 percent Mazclean emulsifier
- 5.0 percent: 5.0 percent d-limonene, 2.5 percent Mazclean emulsifier
- 10.0 percent: 10.0 percent d-limonene, 5.0 percent Mazclean emulsifier

The control consisted of untreated colonies. A 10 percent solution of the Mazclean emulsifier with no d-limonene was also tested.

Efficacy of the treatments was determined 24 hours, 1 week, and 3 weeks after treatment by weighing all surviving ants to determine the effect on workers and brood, and counting the number of living queens to determine queen mortality. Monitoring the surface spray simulation (nest cover on) was ended after 24 hours, at which time it was determined that this method was not as effective as the drench simulation. (See Figure 2 and Tables 1 and 2.) Statistical comparisons among treatments were made by analysis of variance tests and the Newman-Keuls multiple range test.

Field Studies of Dead Ant Efficacy

There were two tests of the effectiveness of Dead Ant in the field: a test by the Environmental Pesticides Group and one by personnel at Texas A & M University.

Environmental Pesticides Group Field Test

Preliminary tests were performed at Mr. Bill Murff's grass farm in Crosby, Texas to evaluate the effectiveness of various concentrations of d-limonene solutions in controlling fire ants in field situations. Four test plots were demarcated, and all mounds within the test plot were flagged. In addition, all mounds within 6 feet of the perimeter of each plot were also marked so that any movement of treated mounds outside the test plot could be detected. Mounds in each plot were treated with a single concentration of d-limonene solution by spraying an even coat of material using a hand-held sprayer. In the larger plots (40 x 50 feet), approximately 2 gallons of solution was sprayed; approximately 1 gallon of solution was sprayed on the smaller plots (10 x 20 feet). After spraying, mounds were treated by drenching with 2 gallons of solution. The size of the plots, the number of mounds in the plot, and the concentration of applied solution was as follows:

- 1.5 percent (+ 0.75 percent Mazclean): 10 x 20 foot plot contained 2 mounds
- 3.0 percent (+ 1.5 percent Mazclean): 40 x 50 foot plot contained 7 mounds
- 5.0 percent (+ 2.5 percent Mazclean): 40 x 50 foot plot contained 6 mounds
- 10.0 percent (+ 5.0 percent Mazclean): 10 x 20 foot plot contained 4 mounds

Treatments were applied on 15 April 1992. These test plots were also used as the source for the product degradation studies.

Texas A & M University Field Test

The test was conducted by Dr. Bastian M. Drees and Mr. Charles L. Barr of the Texas A & M Agricultural Extension Service. The test began on 5 August 1992 on the earthen dam at Lake Somerville, Texas. Plots of land 40 feet wide and of variable length were marked such that each plot contained 10 active fire ant mounds. The plots were grouped into 3 blocks (replication) containing 6 plots each, and treatments were randomly assigned within the blocks. Mound density on the dam was approximately 380 mounds per acre, a density indicative of a population of multiple-queen colonies. Mound activity was determined by lightly disturbing the mound with a pointed wooden tool handle. A mound was considered active if within 15 seconds of disturbance a number of ants came to the surface.

Treatments were applied beginning at 9:00 am and ending at 4:00 pm. The applied solution was mixed in a large drum and dispensed into 2-gallon sprinkler cans. The diffusion nozzle was removed from the end of the spout to give a solid stream permitting better penetration of the mound surface. The following treatments were applied:

- 1.5 gallons per mound of 3 percent d-limonene (with 1 percent Mazclean)
- 1.5 gallons per mound of 4 percent d-limonene (with 1 percent Mazclean)
- 1.5 gallons per mound of 5 percent d-limonene (with 2 percent Mazclean)
- 1.5 gallons per mound of 2 teaspoons Orthene® per gallon of water
- 1.5 gallons per mound of water as a control

Upon mixing with the water, the d-limonene solutions became unusually viscous. This may have been because the water had been sitting in a tank for some period and contained a considerable amount of algae. The thickness of the material reduced its penetration into the mounds, causing it to pool on top of the mounds for some time rather than immediately percolating down. The material did eventually seep down into the mounds.

Post-treatment evaluations began at 9:00 am on 6 August (1 day post-treatment), 7 August (2 days post-treatment), and 12 August (7 days post-treatment). The data were analyzed using an analysis of variance test, and the Newman-Keuls multiple range test was employed to determine significance of differences among treatments (P<0.05).

Product Degradation

Mercury Environmental Services, Pasadena, Texas analyzed the soil samples taken from the test plots on Mr. Bill Murff's grass farm in Crosby, Texas using gas chromatography/mass spectrometry (GC/MS) equipped with a purge trap.

Five point calibrations of d-limonene were run with concentrations at 20, 40, 60, 80, and 100 parts per billion. The GC/MS parameters were 40°C to 220°C with an initial hold time at 2 minutes, ramping at 8°C per minute, with a final hold time of 4 minutes. With these parameters, five sets of samples were analyzed from the four test plots.

Samples were taken from random locations within the plot, but at least 6 feet from the nearest drenched mound. One sample per plot was collected per sample period. The sample plugs were 4.5 x 4.5 x 6 inches deep. Upon collection, samples were placed directly into 1 quart glass jars for transport to the laboratory for analysis.

COST/PAYBACK OF DEMONSTRATION

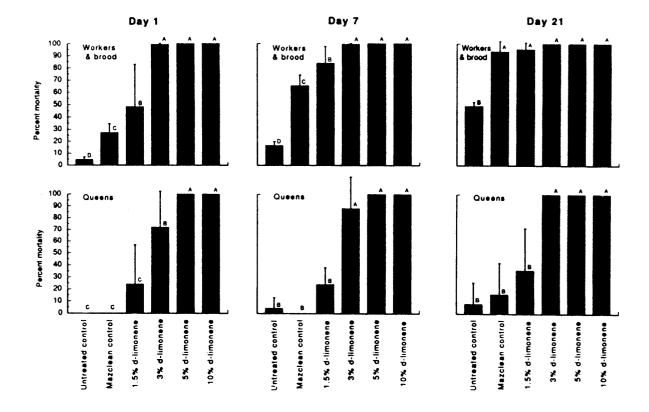
The cost of the demonstration project was \$35,401, with \$25,000 provided by EPA through the Pollution Prevention By and For Small Business Grant Program, and \$10,401 provided by the Environmental Pesticides Group.

RESULTS AND DISCUSSION PERFORMANCE RESULTS

University of Texas Laboratory Evaluation of Dead Ant Efficacy

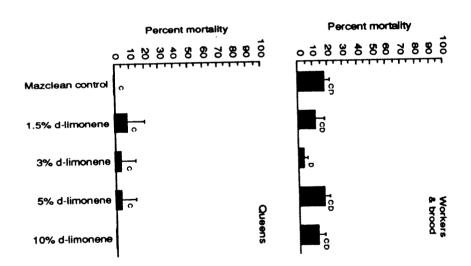
There were highly significant differences in mortality among treatments at all three time periods for both workers/brood (day 1: $F_{10.54} = 65.4$, P < 0.0001; days 7 and 21: $F_{5.29} \ge 98.4$, P < 0.0001; and queens (day 1: $F_{10.54} = 41.0$, P < 0.0001; days 7 and 21: $F_{5.29} \ge 26.2$, P < 0.0001) (see Figure 1 and Tables 1 and 2). Mortality of both workers/brood and queens in the surface spray simulation treatments after 24 hours was not significantly different from the untreated colonies (Figure 3). However, application of the d-limonene solution in the drench simulation, at all concentrations tested, resulted in significant mortality of workers/brood after only 24 hours. There was an obvious concentration effect at the day 1 and day 7 periods, with the 1.5 percent solution being significantly less effective than the three higher concentrations tested, and the 5 percent and 10 percent solutions killing all individuals in the colonies within 24 hours. By day 21, this concentration effect disappeared, and the four dlimonene concentrations did not differ statistically in percent mortality. The Mazclean control also resulted in significant mortality at all time periods. Although there was significantly less mortality than in any of the d-limonene solutions at the day 1 and day 7 periods, on day 21 mortality in the Mazclean-only treatment did not differ significantly from any of the d-limonene treatments, at which time all treatments resulted in over 90 percent mortality. This result suggests that the high concentrations of Mazclean (10 percent) used in the Mazclean-only treatment is toxic to the workers/brood, but that this toxicity is delayed compared to the d-limonene solution.

Effect of D-limonene Solutions on Fire Ant Mortality in Laboratory Colonies. Each colony started with 10 grams of workers and brood (i.e., eggs, larvae, and pupae) and 5 queens. Mortality of workers and brood is shown separately from that of queens for each treatment on each of three evaluation periods. There were 5 replicates of each treatment. Shown are means ± SD. Treatments with different letters on a particular day differed significantly (*P* < 0.05, Newman-Keuls multiple range test). Although only the treatments receiving the drench simulation are shown in this Figure, the analysis of variance tests and multiple range tests included the treatments receiving the surface spray simulation on day 1 (see Figure 3). These data are also presented in Tables 1 and 2 (see section "Tabulation of Data").



Differences among treatments were more pronounced when queen mortality was considered (see Figure 2 and Tables 1 and 2). At all three time periods there was significantly higher mortality in the three highest concentrations of d-limonene than in all other treatments. As with worker/brood mortality, the 5 percent and 10 percent solutions resulted in 100 percent mortality. By day 21, mortality in the 3 percent d-limonene solution also reached 100 percent. There was no queen mortality in the Mazclean-only treatment by day 7, and only slight mortality (16 percent) by day 21, but this was not significantly more than occurred in the untreated control.

Figure 3. Effect of Spray Simulation Application of D-limonene on Laboratory Colonies of Fire Ants 24 Hours After Treatment. Colonies (n=5 for each treatment) had the same composition as those reported in Figure 1. Treatments with different letters differed statistically P < 0.05, Newman-Keuls test). In the statistical analyses, the above treatments were combined with the drench simulation shown in Figure 2. These data are also presented in Tables 1 and 2.



Conclusions

The d-limonene solutions effectively killed the ants within 24 hours after treatment when the liquid was directly applied to all ants through the drench simulation method. The fact that there was only limited mortality in the surface spray simulation application indicates that the material must come into direct contact with the ants to be effective, and that there is no appreciable transfer of material among ants through normal social interactions. When applied directly to the ants, there was a concentration effect with increasing mortality from solutions of 1.5 percent to 3.0 percent to 5.0 percent. There was no difference between the 5.0 percent and 10.0 percent formulations, as both treatments resulted in 100 percent mortality of workers/brood and queens. The measure of queen mortality is the most important, because without a queen the colony will die, whereas as long as there is at least one active queen, she can lay eggs to replace any workers or larvae that might be killed. The Mazclean emulsifier had a delayed effect primarily on the workers and brood, with little effect on the queens. The substantial difference between the efficacy of the dlimonene treatments and the Mazclean-only treatment shows that is the d-limonene, and not the emulsifier, that is responsible for the main effects of the formulation. Environmental Pesticides finds additional support for this conclusion in that the concentration of emulsifier used in the Mazclean-only treatment (10 percent) was twice that of the highest concentration than in any of the d-limonene formulations tested (maximum concentration of Mazclean was 5.0 percent).

Field Test of Dead Ant Efficacy

Environmental Pesticides Group Field Test

Upon thorough examination of all treated mounds on 21 April, 6 days after treatment, no fire ant activity could be detected (Figures 4 through 7). Moreover, no new mounds (i.e., previously unmarked mounds) were observed in or near the treatment plots. Although no formal control mounds were used in this test, several untreated mounds around the perimeter of the test plots showed vigorous signs of fire ant activity upon inspection. All concentrations of the d-limonene solutions appeared to totally eliminate the treated colonies.

Texas A & M University Field Test

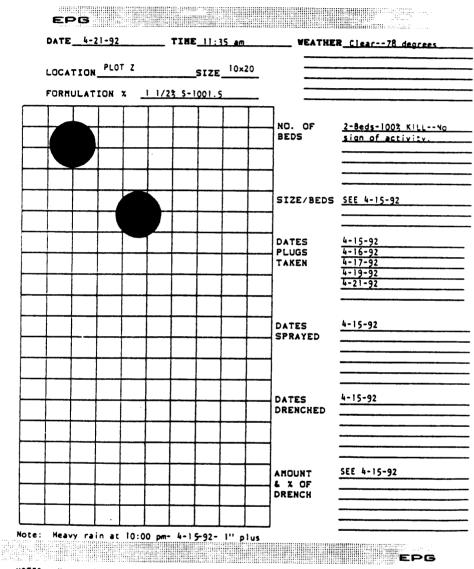
A significant difference was noted among treatments at all three post-treatment test periods ($F_{5,12} \geq 4.92$, P < 0.02) (see Figure 8 and Table 3). At day 1, all treatments containing active ingredient (i.e., all the d-limonene solutions and Orthene®, a class III insecticide known for giving fast effective results in controlling fire ant mounds) rendered a significant percentage of mounds inactive. The 5 percent d-limonene was the most effective of all treatments with 83 percent reduction in the number of active mounds, but this was not significantly more than the other active ingredient treatments. At day 2, all active ingredient treatments still showed significantly more activity than the water control, but activity was also evident in the Mazclean-only treatment, suggesting delayed action of this material. Only the 5 percent d-limonene and Orthene treatments -- which gave 80 percent and 90 percent effectiveness, respectively -- showed significantly more activity than the Mazclean-only treatment. The delayed action of Mazclean was more evident than at day 7, at which

time it showed the third highest activity only behind the 5 percent d-limonene and the Orthene® treatments, although it did not differ significantly from these treatments.

Conclusions

All d-limonene solutions, especially the 5 percent solution, compared favorable with Orthene®. The d-limonene treatments may have been even more effective had the applied material not been so viscous. As with the laboratory studies, there was a delayed but significant effect of the Mazclean applied by itself. However, this effect cannot account for the quick destruction of the fire ant mounds observed in all of the d-limonene treatments. It is the d-limonene that is responsible for the main effects seen in those treatments. These results show that the mortality observed in the laboratory after treatment with d-limonene can be produced in field colonies through mound drenches.

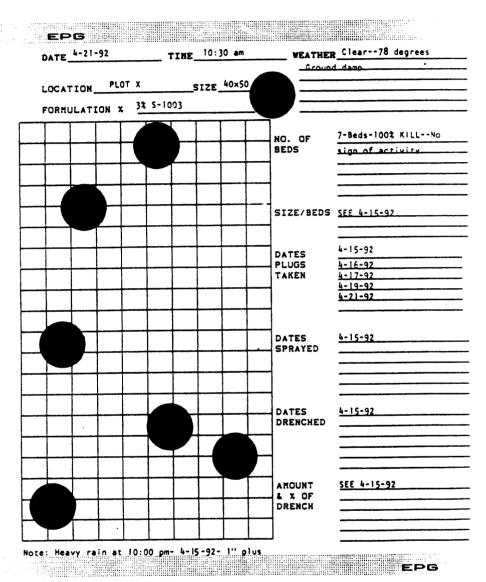
Figure 4. Test Plots Layout and Application Results of 1.5 Percent D-limonene to Fire Ant Mounds at a Grass Farm



NOTED: No new beds within or outside Plot

Light to no yellow effect to grass around drenched beds.

Figure 5. Test Plot Layout and Application Results of 3.0 Percent D-limonene to Fire Ant Mounds at a Grass Farm

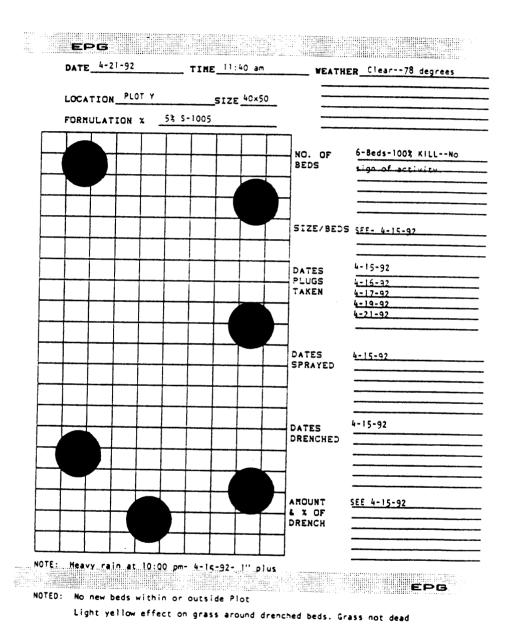


NOTED: No new beds within or outside Plot

Light yellow grass around drenched beds-new growth seen. Grass not dead.

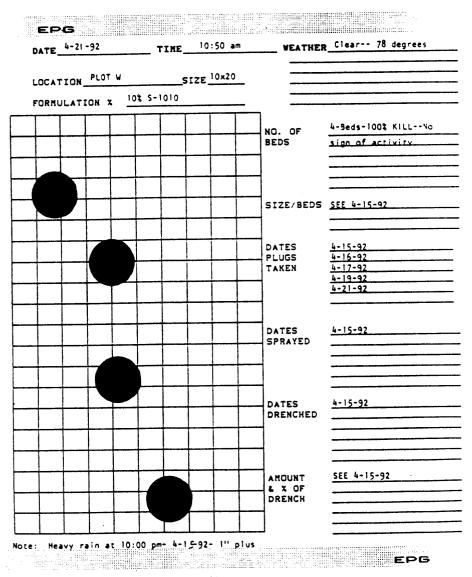
Bed 5- Outside Plot was treated by Probe (24") deep on 4 sides.
the effect was only 75% Kill

Figure 6. Test Plot Layout and Application Results of 5.0 Percent D-limonene to Fire Ant Mounds at a Grass Farm



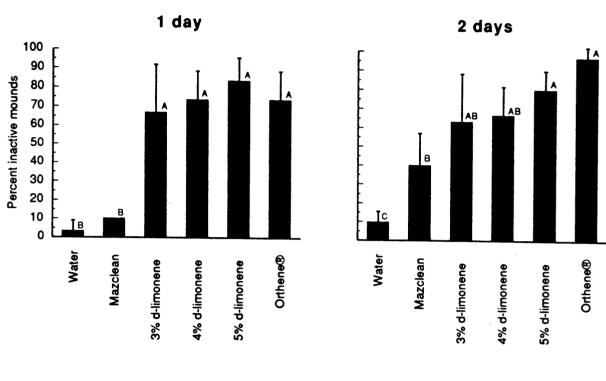
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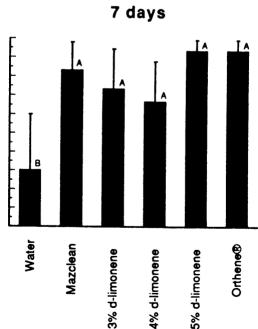
Figure 7. Test Plot Layout and Application Results of 10.0 Percent D-limonene to Fire Ant Mounds at a Grass Farm



NOTED: No new beds within or outside Plot
Grass around drenched beds are yellow, not dead.

Figure 8. Field Tests of Efficacy of D-limonene to Control Fire Ant Mounds. Shown is the percent (\pm SD) mounds out of 10 treated in each plot that were inactive when examined. Three replicates (plots) were made of each treatment. Treatments with different letters on a particular day differed significantly (P < 0.05, Newman-Keuls multiple range test). The data are also presented in Table 3.





Product Degradation

In most plots, there was an initial increase in the amount of d-limonene in the soil samples for the first 2 days after application (see Figure 9 and Table 4), but the product rapidly decreased after this period. By day 6, the quantity of d-limonene had fallen to 0.008 milligrams per kilogram of soil or less in all plots. The reason for the high quantity of d-limonene present of day 1 in the 10 percent plot is not known; even in this case, there was rapid degradation of the product by day 4.

Overall Conclusions

Both in the laboratory and in the field, the 5.0 percent formulation of Dead Ant proved fast and effective in killing fire ants, including workers, larvae, and queens. In the field, its performance was equal to that of Orthene®. As expected, d-limonene rapidly degraded in the environment providing fast, effective; environmentally safe control of fire ants with no long-term residues remaining in the environment after application.

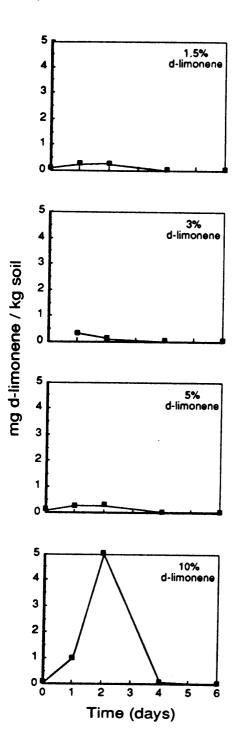
PRODUCT QUALITY VARIANCE

Percentages of d-limonene, emulsifier, and water are very tightly controlled and will comply with the Texas Department of Agriculture and EPA requirements when these are determined. Tolerances are kept within 0.01 percent. Judging from the viscous product that was obtained when mixed with the sitting water in the Texas A & M University test, the purity of the water is important in producing a final product of the proper viscosity. Nevertheless, the thick consistency of the material did not seem to adversely affect the performance of Dead Ant during this test.

CONDITIONS THAT IMPACT PERFORMANCE

Although not specifically investigated, environmental conditions are expected to affect the performance of Dead Ant. Because the product must come into contact with the ants to be effective, the closer the ants are to the surface of the mound, the greater the chances of product contacting all colony members when the mound is drenched. These conditions are found in early afternoon in spring and fall when the ants come near the soil surface to take advantage of the sun-warmed environment of the mound. This may account for the difference in the effectiveness of the field trials done by Environmental Pesticides Group (performed in April when high temperatures were about 75°F) in which 100 percent control was obtained, and those performed by Texas A & M (done in August when high temperatures were about 95°F) in which 5 percent Dead Ant gave about 95 percent control. However, control was effective even in the heat of summer, and excellent results can be expected under most environmental conditions.

Figure 9. Degradation of D-limonene Under Field Conditions. Samples were taken from plots receiving different concentrations of d-limonene sprayed on surface of study plots. The samples were analyzed by GC/MS. The data are also presented in Table 4.



TABULATION OF DATA

University of Texas Laboratory Evaluation of Dead Ant Efficacy

TABLE 1. Effect of D-Limonene Solutions on Worker/Brood Mortality in Laboratory Colonies.

Each colony started with 10 grams of workers and brood. There were 5

Each colony started with 10 grams of workers and brood. There were 5 replicates of each treatment. Treatments with different letters on a particular day differed significantly (P < 0.05, Newman-Keuls multiple range test).

	Percent Mortality (mean ± SD)					
Treatment	1 day		7 days		21 days	
Untreated Control	4.6 ± 2.3 D		16.4 ± 3.5 D		48.8 ± 3.6	В
Drench Simulation						
Mazclean only	27.2 ± 2.4 C		65.4 ± 8.8 C		93.6 ± 8.7	Α
1.5% d-limonene	48.2 ± 34.7	В	83.8 ± 14.0	В	95.4 ± 5.9	Α
3.0% d-limonene	99.4 ± 0.9	Α	99.6 ± 0.6	Α	100 ± 0 A	
5.0% d-limonene	100 ± 0		100 ± 0 A		100 ± 0 A	
10.0% d-limonene	100 ± 0 A		100 ± 0 A		100 ± 0 A	
Surface Spray Simula	ation					
Mazclean only	17.6 ± 4.5 CD					
1.5% d-limonene	12.2 ± 6.0 CD					
3.0% d-limonene	2.6 ± 2.6	D				
5.0% d-limonene	16.8 ± 3.8 CD					-1.1
10.0% d-limonene	13.0 ± 3.9 CD					

TABLE 2. Effect of D-Limonene Solutions on Queen Mortality in Laboratory Colonies. Each colony started with 5 queens. There were 5 replicates of each treatment. Treatments with different letters on a particular day differed significantly (P < 0.05, Newman-Keuls multiple range test).

	Percent Mortality (mean ± SD)				
Treatment	1 day	7 days	21 days		
Untreated Control	0 C	4.0 ± 8.9 B	8.0 ± 17.9 B		
Drench Simulation					
Mazclean only	0 C	0 B	16.0 ± 26.0 B		
1.5% d-limonene	24.0 ± 32.9 C	24.0 ± 43.4 B	36.0 ± 35.8 B		
3.0% d-limonene	72.0 ± 30.0 B	88.0 ± 26.8 A	100 ± 0		
5.0% d-limonene	100 ± 0 A	100 ± 0	100 ± 0		
10.0% d-limonene	100 ± 0 A	100 ± 0 A	100 ± 0		
Surface Spray Simula	ition				
Mazclean only	0 C				
1.5% d-limonene	8.0 ± 11.0 C				
3.0% d-limonene	4.0 ± 8.9 C				
5.0% d-limonene	4.0 ± 8.9 C				
10.0% d-limonene	0 C				

Field Tests of Dead Ant Efficacy: Texas A & M University Field Test

TABLE 3. Field Tests on the Efficency of D-Liomene Solutions to Control Fire Ant Mounds.
Shown is the percent (± SD) mounds out of 10 treated in each plot that

Shown is the percent (\pm SD) mounds out of 10 treated in each plot that were inactive when examined. There were 3 replicates (plots) of each treatment. Treatments with different letters on a particular day differed significantly (P < 0.05, Newman-Keuls multiple range test).

	F	Percent Mounds Inactive					
Treatment	1 day	2 days	7 days				
Water control	3.3 ± 5.8 B	6.7 ± 5.8 C	30.0 ± 30.0	В			
Mazclean only	10.0 ± 0.0 B	40.0 ± 17.0 B	83.0 ± 15.0	Α			
3.0% d-limonene	66.7 ± 25.0 A	63.0 ± 25.0 AB	73.0 ± 21.0	Α			
4.0% d-limonene	73.0 ± 15.0 A	66.7 ± 15.0 AB	66.7 ± 21.0	Α			
5.0% d-limonene	83.0 ± 12.0 A	80.0 ± 10.0 A	93.0 ± 5.8	Α			
Orthene®	73.0 ± 15.0 A	96.7 ± 5.8 A	93.0 ± 5.8	Α			

Product Degradation

TABLE 4. Degradation of D-Limonene under Field Conditions.
Samples were taken from plots treated with one of four concentrations of d-limonene. Analysis was performed by GC/MS.

	Quantity of D-limonene (mg/kg soil)					
Concentration	Day 0	Day 1	Day 2	Day 4	Day 6	
1.5%	0.053	1.240	0.288	0.010	0.002	
3.0%		0.309	0.125	0.020	0.008	
5.0%	0.088	0.237	0.265	0.010	0.007	
10.0%	0.036	0.983	5.000	0.070	0.002	

COST/BENEFIT ANALYSIS

The above results show that d-limonene is as effective as a leading toxicant, Orthene®, in controlling fire ants. Currently available information indicates that all Dead Ant ingredients are non-toxic to humans and other vertebrates and are not harmful to the environment. In addition to being safe and effective, Environmental Pesticides Group expects to offer Dead Ant at a price competitive with Orthene® and other leading fire ant control products. The approximate retail cost of Orthene® is \$0.25 to treat a single mound. Currently, Dead Ant can be produced at about \$0.12 per mound treatment (1 to 1.5 pints per 6- to 8-inch mound), and the anticipated retail price is \$0.22 to \$0.25 per mound treatment.

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Incentives

The product is almost ready to be introduced into the marketplace. The major hurdle remaining is EPA registration of the product, which requires extensive tests on animal toxicity and environmental fate.

The targeted markets will include farmers, nursery and sod growers, park maintenance personnel, pest control operators, gardeners, and home owners -- all those concerned with the control of fire ants.

Barriers

The only shortcoming is an occasional, temporary yellowing of surrounding grass following treatment of mounds. Environmental Pesticides Group is testing different concentrations of Dead Ant under various conditions (e.g., grass type and temperature) in order to find the combination of conditions that will eliminate grass discoloration and still provide effective fire ant control.

POLLUTION PREVENTION IN CADMIUM PLATING

by

Mandar Sunthankar IonEdge Corporation Fort Collins, CO 80526

ABSTRACT

Cadmium is electroplated on many industrial components because of its desirable lubricity and corrosion resistance properties. However, the use of cyanide baths in electroplating and toxic waste disposal related to cadmium are of significant environmental concern. In recent years, 50/50 zinc-cadmium alloy coating has shown promise as an alternative to cadmium. This alloy uses 50 percent less cadmium in plating and exhibits corrosion properties superior to those of cadmium.

To minimize environmental and occupational hazards related to cadmium electroplating, a novel dry plating technique has been developed by the lonEdge Corporation. This plating eliminates liquid chemicals and prevents solid waste using *in situ* reclaim. The dry plating process is suitable for plating the 50/50 zinc-cadmium alloy. In order to explore the commercial potential of this dry plated alloy, its lubricity was studied and compared with that of the electroplated cadmium.

In the research conducted, the 50/50 zinc-cadmium alloy was dry plated on mild steel washers. Sample washers were also electroplated for comparison. These washers were used as substrates for the lubricity study. The lubricity was measured in terms of coefficient of friction using pin-on-disk method. The chemical composition of the dry plated alloy was determined using Energy Dispersive Spectrometric (EDS) x-ray analysis. The data indicated that the coefficient of friction of dry plated 50/50 zinc-cadmium alloy is 0.133 compared to 0.127 for electroplated cadmium. The statistical t-test of significance predicts that this difference is not significant. As a result, lonEdge concluded that the lubricity of this alloy is competitive with that of cadmium and superior to that of known values of zinc. This lubricity study indicates promising commercial potential for the dry plated 50/50 zinc-cadmium alloy.

INTRODUCTION

PROJECT DESCRIPTION

Electroplated cadmium is preferred in many large-scale fastener applications because of its superior lubricity and corrosion resistance in marine environments compared to that of other coatings.¹ In cadmium electroplating operations, exposure to toxic cadmium dust is a safety issue, and disposal of cadmium sludge is an environmental issue.² Consequently, there is substantial interest in reducing the use of cadmium and related waste. However, two decades of extensive effort in this area has yet to result in an effective economical solution.

The dry plating process developed at the lonEdge Corporation was proposed as exhibiting promising pollution prevention potential. Dry plating eliminates use of liquid chemicals and prevents solid waste using *in situ* reclaim. In recent years, a 50/50 zinc-cadmium alloy has been qualified by the automobile industry for some fastener applications.³ Environmentally safer zinc plating alone does not exhibit the frictional property required to replace cadmium. The frictional coefficient of zinc (0.16) is relatively higher than that of cadmium (0.123). The addition of cadmium is likely to enhance the lubricity of zinc and bring it into an acceptable range for fasteners. The proposed effort uses a non-liquid chemical process for plating this lower-friction zinc-cadmium alloy and studies its frictional property with respect to cadmium.

A study of the lubricity of a dry plated 50/50 zinc-cadmium alloy is desirable for its potential application in a variety of industries. The following objectives were targeted for this research:

- Measure the lubricity of dry plated 50/50 zinc-cadmium alloy in terms of coefficient of friction.
- Compare the coefficient of friction of dry plated 50/50 zinc-cadmium with that of electroplated cadmium.

Outline of Process/Product

Cadmium is a known toxic material. Occupational exposure to cadmium dust and vapors is a safety issue, and disposal of cadmium is an environmental issue.² Most cadmium electroplating is carried out in cyanide and other toxic chemical baths. Alternative coatings of zinc or aluminum do not exhibit certain unique combinations of properties of cadmium.^{4,5} Consequently, despite escalating costs of waste management from stricter environmental regulations, cadmium electroplating continues to be widely used in defense, aerospace, and automobile industries.

There is substantial interest in reducing cadmium in plating.² A new coating recently qualified by the Ford Motor Company is a 50/50 zinc-cadmium alloy³, (i.e., 50 percent by weight zinc in cadmium (Ford Specification S54-M). This alloy contains 50 percent less cadmium in deposited coatings. The 50/50 zinc-cadmium alloy shows suitable combination of properties of both zinc and cadmium, and is superior to cadmium in corrosion properties.³ The outdoor exposure studies of competing coatings in various environments indicated that zinc-cadmium alloys outperform other coatings in every environment tested.⁶ The lubricity properties of zinc-cadmium alloys were not reported.

Various compositions of the zinc-cadmium alloy coatings are currently applied using the mechanical barrel plating process.^{3,6} This plating is a liquid chemical operation free of cyanide and chelates.⁷ However, significantly larger amounts of cadmium and other toxic chemicals are discharged following each batch of plating compared to electroplating. This results in a cost intensive operation.⁸

A plating method that minimizes the use of cadmium as well as the use of toxic liquids is desirable. The dry plating addresses this need with three unique features:

- The plating rate is competitive with electroplating.
- Solid waste is minimized using in situ reclaim and recycle.

Furthermore, the zinc-cadmium alloy would reduce the amount of cadmium released into the environment by discarded components.

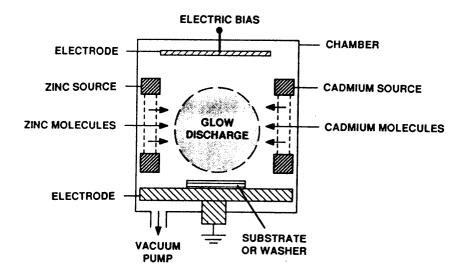
Unique Features of the Proprietary Dry Plating Process.

In the dry plating of zinc-cadmium alloy, simultaneous plating of zinc and cadmium species is conducted under a neutral gas glow discharge condition. The glow discharge offers the following advantages:

- The collision of ionized neutral gas molecules with zinc and cadmium particles results in atomic mixing of the species before plating.
- The bombardment of the ionized species on the substrate enhances adhesion of the growing layer to the substrate.
- Energetic particle bombardment of the surface densifies the alloy layer.

Details of this process cannot be disclosed due to the proprietary nature of this method. A schematic of the setup is shown in Figure 1. The scattered, non-directional nature of the plating molecules produces excellent plating uniformity without rotation of the parts. In dry plating, surface cleaning before plating is performed in the same process cycle using neutral gas ion bombardment. There are no by-products or waste generated in this "dry" cleaning. In addition, build-up of coating at edges and corners is avoided. Plating rates competitive with electroplating have been accomplished in the dry plating process. Typical cycle time for a 0.5 mil-thick plating is less than 10 minutes.

Figure 1. Dry Alloy Plating Apparatus



Summary of Prior Studies Conducted

Preliminary studies, such as salt fog corrosion test (ASTM B117), adhesion test (ASTM B571), and solderability test using pull method, conducted on dry plated cadmium coatings have been promising. In addition, other studies have been completed under the Small Business Innovative Research (SBIR) program. In a study sponsored by the National Science Foundation (NSF), it was demonstrated that the dry plating rate can be varied over nearly two orders of magnitude, from 0.01 mil per minute to 0.7 mil per minute. In a Department of Defense-sponsored study, it has been demonstrated that the plating uniformity on complex shapes is competitive to electroplated counterparts. This uniformity also surpassed required military standards according to specification MIL-C-8837B.

Technology Advantages

The extraneous metal deposits in the dry plating chamber will be continuously or periodically recovered or transferred to the metal source itself using a proprietary technique.

Occupational exposure of an operator to hazardous dust and fumes is an issue of concern in a plating operation. The dry plating process eliminates this exposure. A study was also conducted according to ASTM D4185 using Atomic Absorption Spectrometry (AAS) to determine the level of cadmium or zinc dust emission from the dry plating equipment. This study showed that the level of cadmium and zinc emission was below the AAS detection limit (i.e., below 50 parts per billion: 10⁻⁹ grams per cubic meter). This is comparable to the levels of these metals measured in non-polluted air. In addition, dry plating eliminates waste and sludge disposal.

APPLICATION

Process/Product Replaced

There are 1,166 facilities in the U.S. predominantly electroplating cadmium.² The large industry of cadmium platers and users is well established with decades of proven procedures and specifications. This industry requires new yet compatible and economical methods of waste reduction. Electroplated cadmium is preferred when users need corrosion protection in a marine environment, or when coating lubricity is of prime concern for components like steel fasteners.¹ For example, Ford Motor Company uses 30 million cadmium-plated nuts annually in addition to millions of other components; 50,000 to 60,000 pounds of components are cadmium-plated daily in other plating shops.³ Electroplated cadmium typically used is under 0.5 mil (13µm) thickness.

Current Cadmium Plating Methods

Three distinct methods are used in plating cadmium or zinc under 1 mil thick: electroplating, mechanical plating, and vacuum cadmium. Cyanide bath electroplating has been the overwhelming choice for most applications because of good quality and high throughput at reasonable cost. Mechanical plating is limited to barrel plating of small components, and is more competitive for thicker coatings (>0.5 mil). Vacuum cadmium is expensive and is preferred only when hydrogen embrittlement has to be

eliminated in some defense and aerospace applications. Barrel plating is used in plating small parts, such as fasteners in bulk (2,000 to 10,000) in a rotating barrel. Rack plating is used for individually plating larger parts on racks.

Occupational Hazards of Cadmium

Occupational exposure to cadmium primarily occurs through inhalation of dust or fumes.² According to EPA's 1984 guidelines, cadmium is classified as a group B1, probable human carcinogen.² Current OSHA permissible limits for 8 hour cadmium fume exposure is 0.1 milligrams per cubic meter and that for cadmium dust exposure is 0.2 milligrams per cubic meter (29 CFR 1910.1000). The current EPA standard for limiting the amount of direct cadmium discharge in wastewater on a monthly average is not to exceed 0.26 milligrams per liter (40 CFR 433).

Other Environmentally Safer Coatings

Zinc exhibits many properties similar to cadmium. Zinc plating is relatively inexpensive and has been used to replace cadmium where appropriate. However, as can be seen from Table 1, cadmium has better lubricity and torque coefficient and is more corrosion resistant in marine environments.

TABLE 1.	Comparison of (Cadmium and Zinc	Plaging Characteristics	3
	<u> </u>			_

Characteristic	Cadmium Plating	Zinc Plating	
Typical thickness	0.2-0.5 mil	>1.0 mil	
Better corrosion resistance in:1	Marine environment	Industrial environment	
Corrosion product ¹	Thinner, ductile, adherent	Thicker, harder, loose	
Coefficient of friction ^{1,11}	Lower than steel (0.12)	Higher than steel (0.16)	
Lubricity ¹⁰	Excellent	Fair	
Torque coefficient ¹⁰	0.15-0.25	0.21-0.33	

Low torque coefficient is preferred especially in automated fastener assembly operations. In this regard, the lubricity of dry plated 50/50 zinc-cadmium alloy is likely to be superior to that of zinc and closer to that of cadmium.

A study conducted by the U.S. Army indicated that the combination of corrosion resistance and lubricity of cadmium was superior to 14 other competing materials including aluminum. According to two other studies conducted by the U.S. Air Force, aluminum is useful at higher temperatures and is superior in abrasion resistance. In such applications, aluminum could replace cadmium. However, for equal thickness, cadmium performed better in salt fog corrosion tests. Close tolerance in fastener applications require thinner, ductile coatings of cadmium. Electrically non-conductive corrosion products of aluminum are unacceptable in the

electrical connector market. The complexity in aluminum plating operation and high capital cost made it unlikely that it will ever become common in plating shops.¹³

Wastes Prevented

Conventional electrochemical plating processes use several hazardous liquid chemicals in large quantities. These include alkaline cyanide baths, and various chlorides, sulfates, phosphates, and fluoborates of heavy metals. On the contrary, dry plating does not use any liquids. In summary the dry plating method offers the following benefits in that it is a simple plating operation; recycles pure solid metal *in situ*; eliminates hazardous liquids used in plating; eliminates subsequent wastewater treatment and sludge; reduces costs related to chemical equipment; and reduces capital costs and operating space. Since no liquids are required in plating, the subsequent costs and liabilities of waste treatment, disposal, and effluent discharge are eliminated.

<u>Cadmium Reclamation from Discarded Components</u>

In cadmium plating shops, plated components that are rejected for poor quality are stripped in toxic liquids before replating. Instead, dry plating equipment can be used to reclaim this cadmium as well as that from used discarded components before incineration. Contaminated cadmium reclaimed this way can be recycled by the cadmium producers.

Cross Segment Uses

The initial niche of dry plating technology is likely to be in steel fasteners and electrical connectors used on a large scale in the Department of Defense and the automobile industry. Current trends in these segments indicate increasing use of alternatives to cadmium electroplating. Another potential segment for this technology is likely to be the high strength steel components used in the defense and aerospace industry, because dry plating will eliminate hydrogen embrittlement and the process will be very competitive. Available information suggests that among high strength steel users, the U.S. Navy and Air Force are leading the effort in seeking alternatives to cadmium electroplating.

Dry plating will also plate zinc without using liquid chemicals. As an alternative to zinc electroplating (galvanizing), dry plating has potential in strip-steel plating and wire coating. Considering the high rates of dry plating, galvanizing steel sheets and wires may be done economically. Dry plating also can galvanize sheet metal and wire products after metal finishing.

PROCEDURE

DEMONSTRATION PROCEDURES

lonEdge used two independent experimental procedures to complete this project: (1) the alloy plating plus composition analysis; and (2) the lubricity measurements. The 50/50 zinc-cadmium alloy deposition process needed some improvements so that the same composition could be plated repeatably. In this

development phase, smaller-sized mild steel substrates were plated so that these could be placed in composition analysis equipment. The composition analysis was performed using Energy Dispersive Spectrometric (EDS) x-ray analysis. Later, larger, medium carbon mild steel washers were plated for lubricity study. Washers are frequently used for frictional (lubricity) tests and are commercially available in various sizes. Lubricity was measured on a pin-on-disk frictional test set-up. The pin and the disk (washer) are made of the materials to be tested against each other. In this case, the interest was in the performance of the alloy coating against mild steel. Consequently, the washer was alloy plated and the pin was made of mild steel.

The Dry Plating Apparatus

The basic principle of the alloy plating process is as follows: A desired composition of the alloy is obtained by proportioning the quantity of zinc and cadmium species injected in the chamber. This quantity is proportional to the amount of source material to be plated. To minimize complexity in the process, the source temperature is maintained nearly constant to allow for better control over the process and produces acceptable repeatability. The desired composition is obtained by keeping the injected quantity of zinc species constant while varying the injected quantity of cadmium species. The process parameters are manipulated to vary the amount of metal species in the injected stream. The plated samples are EDS analyzed. This exercise helped to understand the performance of the plating process and its sensitivity to the process conditions. IonEdge cannot disclose further details of this process due to its proprietary nature.

The glow discharge apparatus used for dry plating is made by Technics (Model Hummer V). The minimum pressure obtained by a 1.5 horsepower mechanical pump in this apparatus is about 20 milliTorr. Zinc and cadmium sources were 99.5 percent pure and were purchased from a commercial vendor.

Sample Preparation for Alloy Plating Tests

Medium carbon steel substrates were used to simulate typical practical conditions where cadmium coatings are used. For the initial composition experiments, the substrates were made from available commercial grade, cold-rolled mild steel, 0.195 centimeters thick. These were cut into approximately 1.15 centimeter squares and ultrasonically degreased in acetone and methyl alcohol. All substrates were polished on 600 grit SiC sandpaper and subsequently polished to mirror finish using 0.3 micron alumina slurry. This same degreasing and polishing procedure was used for as-purchased mild steel commercial washers.

EDS X-ray Analysis

This quantitative chemical analysis was performed in the Scanning Electron Microscope (SEM) by measuring the energy and intensity distribution of the x-ray signal generated by a focused electron beam. A Phillips 505 SEM with secondary backscatter and windowless x-ray detector, and KeVex Super 8000 EDS spectrometer with associated Quantex software was used. During measurements, the incident electron beam was set at 15 degrees to the normal to the substrate, and the x-ray detector sensed characteristic x-rays emitting from the surface. To minimize measurement error due to microscopic variations in composition of inhomogeneous

zinc-cadmium alloy, a 0.2 x 0.2 millimeter area was scanned and quantitative measurements were averaged.

The Lubricity Test Set-up

The pin-on-disk set-up consists of a balanced beam (arm) at the end of which a mild steel pin is mounted in a bushing. This pin rests on a washer mounted on a cylindrical aluminum block. The washer is plated with the desired coating. There is zero weight on the pin before the start of the test. A known weight is placed on the pin as a normal load. The washer is turned by a motor (Danfoss, Model Varispeed 2000) at a desired speed (typically 100 to 1,000 revolutions per minute). The frictional force generated at the contact between the pin and the coating is displayed on a bridge amplifier/meter (Ellis Associates). The test is run for a few hundred to 10,000 rotations (cycles) depending on the strength of the materials being tested.

Sample Preparation for Lubricity Study

The mild steel pin was made in-house from a 0.625 centimeter diameter stock material. The tip of this pin was machined to a hemispherical shape using a cutting tool, and the tip was polished to mirror finish on a buffing wheel using 0.3 micron alumina slurry. The mild steel washers selected for the lubricity test were 3.75 centimeters in diameter and 2 millimeters thick. These washers were degreased and ground to an acceptable flatness and polished on a 600 grit SiC sandpaper. These were plated with the desired material and mounted on a 5 centimeter diameter and 5 centimeter long aluminum cylindrical block during tests. Five polished washers were sent to Denver Metal Finishing Company, Denver, Colorado, for conventional bright cadmium electroplating. The lubricity of these was compared later with that of the dry plated washers.

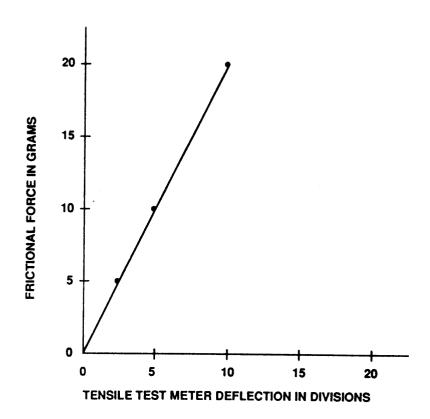
Pin-on-disk Apparatus Calibration

According to the standard calibration procedure, the weight on the pin due to the balanced beam before calibration was zero (i.e., the arm movement was nearly free of weight and friction around the pivot. The tester was calibrated using standard weights in grams. During dynamic frictional studies, the frictional force at the interface of pin and washer created a drag (load) that resulted in minute tension and bending of the arm. This bending induced by the load, called frictional force, is proportional to the friction coefficient. A sensitive tensile test meter indicated the extent of bending in terms of divisions on an arbitrary scale. This bending can also be induced using standard weights in static mode. The scale was correlated to standard weights that indicate frictional force. Accordingly, standard weights were applied to the arm and a calibration curve (Figure 2) was developed for the apparatus. Using this curve, the frictional force was extrapolated from deflection of the meter during dynamic tests. The formula for Coefficient of Friction is:

Coefficient of Friction = Frictional Force Normal Load

where Normal Load is placed on the pin during the dynamic test.

Figure 2. Calibration Curve, Deflection versus Frictional Force



Frictional tests were run without using a lubricant to simulate typical situations for cadmium plated fasteners. In the case of hard coatings, such as diamond, nitrides, and carbides, weights in excess of 200 grams typically are used as normal loads on the pin, and washer rotational speeds are in hundreds of revolutions per minute. For soft surfaces like cadmium and mild steel, this procedure was too severe. The metal was immediately seized, gouged, and penetrated.

Consequently, the following acceptable procedure was developed over a series of experiments. The frictional values were compared to those published. A 20 gram weight was placed on the pin as normal load. The washer was turned by a motor at 50 revolutions per minute. The frictional force generated at the contact between the pin and the washer surface was displayed on the tensile test meter. This test was run for 100 rotations, or 2 minutes, on the washer. Longer time or larger loads were not possible for most samples. In the case of fastener applications, only a few turns are required for tightening. Consequently, 100 turns should provide adequate simulated data for multiple fastening of the parts.

Cost of Demonstration

The research portion of this project was conducted in the Materials Laboratory at Colorado State University (CSU) according to a prior agreement. The total cost of the project, including labor, overhead, materials, and equipment was \$27,025, with \$25,000 provided by EPA through the Pollution Prevention By and For Small Business Grant, and the remaining \$2,025 provided by IonEdge. The direct labor included the Principal Investigator and one Mechanical Engineering undergraduate as a part-time technical assistant. All necessary equipment was located at CSU and was made available for research, thus there were no costs to use these items except for repairs. For accurate EDS data and analysis, the zinc-cadmium alloy-plated test samples were sent to an outside analytical service under subcontract.

The time required to develop a procedure to adjust the ratio of zinc and cadmium in the alloy was approximately six weeks longer than the projected two months due to high sensitivity of cadmium species to the process parameter variations. This problem was corrected by regulating the supply of cadmium species entering the chamber. The lubricity study was completed in the scheduled two months. During the last month of experiments, the anode power supply in the glow discharge unit broke down due to auto-transformer overload. This required repairs and caused a three week delay. Total time to complete this project was approximately six months.

RESULTS AND DISCUSSION

PERFORMANCE RESULTS

50/50 Zinc Alloy Plating

These plating experiments were conducted on smaller size, 1.15 square centimeter mild steel substrates to avoid sectioning and excessive handling of washers. The smaller substrates could be easily placed inside the SEM chamber. The EDS x-ray composition analysis indicated that zinc content was high initially (zinc = 62 percent by weight) as indicated by Figure 3, sample #7. As the cadmium flow was increased by changing process parameters, a larger than expected shift in favor of cadmium content in the alloy (cadmium = 77 percent by weight) was noticed for sample #E2-4. This EDS data is presented in Figure 4. The process parameters were adjusted in successive experiments and composition was analyzed. The quantity of cadmium in the alloy was lowered to 49 percent as shown in Figure 5 of sample #E2-11.

Figure 3. EDS Analysis of Dry Plated 62/38 Zinc-Cadmium Alloy

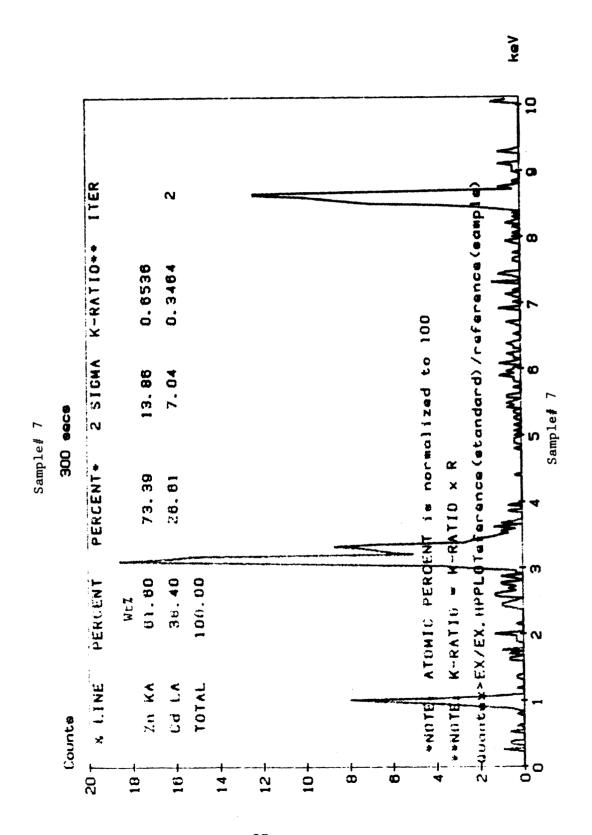
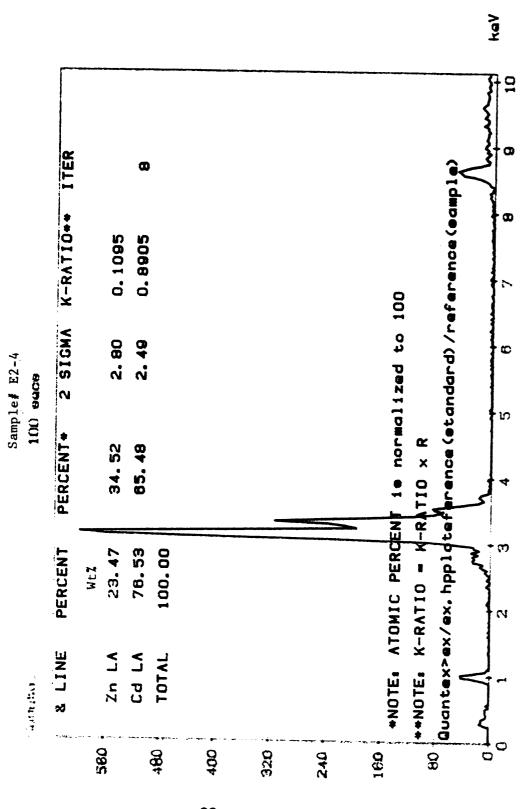
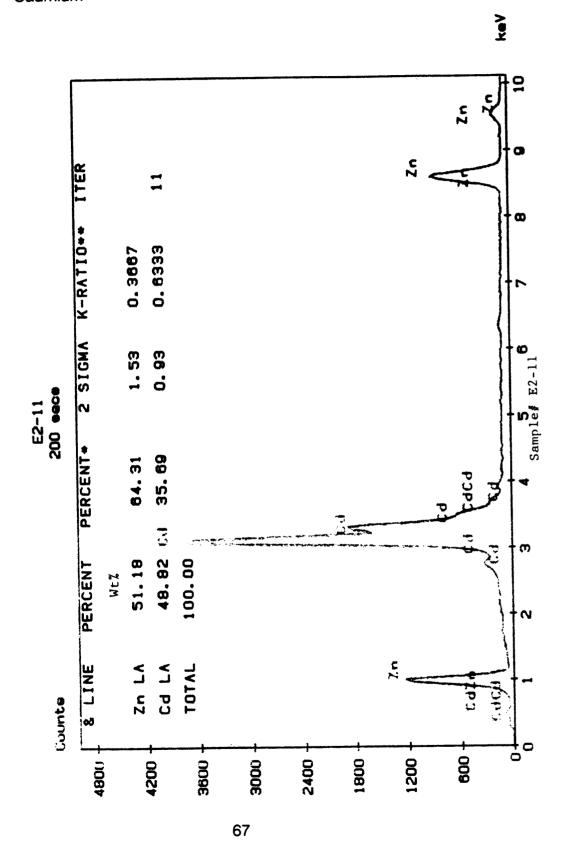


Figure 4. EDS Analysis of 77 Percent Cadmium, Dry Plated Zinc-Cadmium Alloy



Sample# E2-4

Figure 5. Dry Plated Zinc-Cadmium Alloy with 50 Percent (± 2 Percent) Zinc or Cadmium



The composition of alloy dry plated on sample #E2-11 being within acceptable limit (\pm 2 percent) of the 50/50, it was concluded that the desired zinc-cadmium alloy was dry plated.

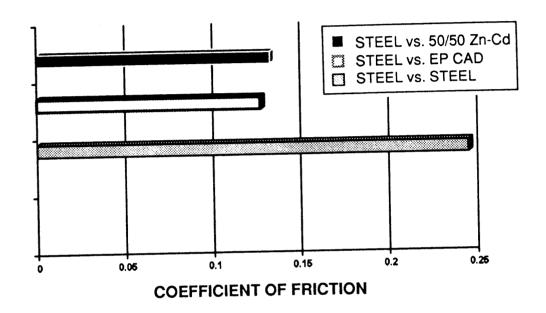
The Lubricity Study

Three washers were also dry plated with the 50/50 zinc-cadmium alloy under the same process conditions. Comparative lubricity studies were conducted on plain steel washers, cadmium electroplated washers, and 50/50 zinc-cadmium dry plated washers. Lubricity was measured in terms of coefficient of friction as described earlier. The average of frictional measurements for electroplated cadmium (0.127) was quite close to that reported in the literature (0.123). This indicated that the error in the measurement or procedure was relatively small. This low value of cadmium frictional coefficient is preferred in the fastener industry. The other data presented in Table 2 indicate that the frictional coefficient of the dry plated 50/50 zinc-cadmium alloy is slightly higher than that of electroplated cadmium. However, this difference is statistically not significant (as explained later) and is likely to be in the range of acceptable values for steel fasteners. This result is presented in Figure 6 using a bar graph.

TABLE 2. Lubricity of Mild Steel Washers, Cadmium Plated Washers, and Alloy Plated Washers

Washer Type	Sample #	Deflection (Divisions)	Frictional Force	Frictional Coefficient	Average Coefficient
No coat	PS-1	3.4	6.8	0.34	
No coat	PS-2	1.5	3.0	0.15	0.245
EP Cad	DM-2	0.8	1.6	0.08	
EP Cad	DM-3	1.0	2.0	0.10	
EP Cad	DM-4	2.0	4.0	0.20	0.127
Zn-Cad	50Cd-1	1.0	2.0	0.10	
Zn-Cad	50Cd-2	2.0	4.0	0.20	
Zn-Cad	50Cd-3	1.0	2.0	0.10	0.133

Figure 6. Bar Graph Showing Lubricity of Mild Steel Washers, Cadmium Plated Washers, and Alloy Plated Washers



PRODUCT QUALITY VARIANCE

The appearance of the dry plated substrate surface was dull metallic gray compared to the bright metallic of the electroplated counterparts. Two factors were likely to influence this appearance. In the electroplating process, brighteners are added to the bath to give the part surface added shine. Second, the morphology of the dry plated surface is granular and consequently non-specular, as the SEM micrograph indicates. A small degree of preferential crystal growth appears to have occurred during plating. This is not uncommon in low pressure processes and can be corrected with process modifications. However, the grainy surface of this ductile metal is not likely to influence the frictional properties because the surface is smeared easily as soon as the test begins and the structure is altered. This is also true with the electroplated cadmium surface.

Significant variation in frictional values of plain polished steel washers was attributed to the variation in hardness from washer to washer. When the material hardness was relatively lower, the wear track was abraded and galled, and the friction was greater. The debris could be observed on the tip of the pin under the microscope. When the material was harder, little debris was observed, and the wear track was less damaged. On the other hand, the wear track on the electroplated cadmium, as well as on the dry plated alloy washer, was also damaged soon after the test was resumed. Similar debris could be observed on the pin tip. However, friction was low and its variations were relatively small due to the soft and ductile nature of these materials. In other words, lubricity was much better, and seizing was prevented. This property is valuable for fasteners that need consistent and lower torque values in automated assembly operations.

The statistical information in Table 3 compares the coefficient of friction of dry plated and electroplated samples.

TABLE 3. Statistical Information Regarding Coefficient of Friction of Dry Plated and Electroplated Samples

	Dry Plated 50/50 Zn-Cd	Electroplated Cadmium
Mean	$x_1 = 0.133$	$x_2 = 0.127$
Standard Deviation	$s_1 = 0.058$	$s_2 = 0.064$
Variance	$s_1^2 = 0.003$	$s_2^2 = 0.004$
Sample Size	$n_1 = 3$	$n_2 = 3$

The variation is frictional values of dry plated samples is slightly smaller than the electroplated counterpart. In order to estimate if the difference in the mean would be statistically significant for the large population based on the small sample size, the t-test of significance was conducted. This analysis indicates that, at 99 percent confidence level, there is no statistically significant difference between the lubricity of dry plated 50/50 zinc-cadmium alloy and electroplated cadmium. IonEdge concludes that the dry plated 50/50 zinc-cadmium alloy has the potential to replace electroplated cadmium.

CONDITIONS THAT IMPACT PERFORMANCE

The lubricity, or frictional coefficient, of a material is a surface phenomena. Consequently, surface contamination could influence the lubricity property. Similarly, changes in physical properties of materials, such as hardness and crystal structure, could result in changes in lubricity. Soft materials like cadmium, zinc, and graphite are relatively less sensitive to these factors except for severe surface contamination. For example, zinc oxide is a corrosion product of zinc that accumulates on the metal's surface after a period of exposure to the environment. This oxide has poor lubricity, which is unacceptable in the fastener industry. Consequently, zinc coating is avoided for critical fastener applications.

In the case of dry plating, pure metals are plated, and surface contamination is avoided due to vacuum environment. Also, absence of liquids in dry plating will prevent surface or bulk contamination that can occur in electroplating. The other factor affecting lubricity is the zinc to cadmium ratio in the plated alloy. If the composition is held within \pm 2 percent of the desired value, the lubricity change is not likely to be noticeable.

COST/BENEFIT ANALYSIS

The following data present waste generated and costs incurred in the electroplating operation that is targeted for elimination using the dry plating process.

Costs Due to Environmental Regulations

Cadmium electroplating operations use large amounts of chemicals and water. A typical job-shop discharges between 60,000 to 150,000 gallons of effluents per day, and may use over 20,000 pounds of cadmium per year. In order to comply with EPA regulations, the installation cost for water pollution control system and associated hardware exceeds \$650,000. Furthermore, the daily cost of wastewater treatment in a typical medium size facility is approximately \$1,200 per day including the cost of 15 to 20 tons of toxic sludge disposal per week. As lower discharge limits are being set on heavy metals and as the number of sludge disposal sites is diminishing, the cost of waste treatment is escalating.

Anticipated Waste Reduction Cost Benefits

Waste treatment costs in cadmium electroplating are outlined in Table 4. These are typical estimates of a few job shops studied. These costs will be eliminated by the dry plating process. Cadmium waste generated, if any, will be in solid metal form that can be reclaimed and recycled.

TABLE 4. Electroplating Waste Treatment Costs⁴

Cost Factor (hourly)	Cost of Waste Treatment Facility
Recurring	\$86
Labor	38% - maintenance, analysis, records
Chemicals	16% - cyanide treat, Cd precipitation
Disposal	17% - haz. wastes, Cd sludge, filters
Utilities	26% - water, electric
Miscellaneous	3% - OSHA compliance, permits, insurance
Capital	\$42 (7 year amortized; 2,000 hours/year)
Total Cost	\$128/hour or approx. \$1,000/day/facility/shift

Anticipated Labor Cost Savings in Dry Plating

The dry plating operation will use the same process flow as that of vacuum cadmium plating. The significant difference, however, will be in labor cost savings as shown in Table 5. The dry plating cycle being competitive with electroplating in deposition rate and time, labor costs in the actual plating cycle were assumed to be the same. As the data suggest, dry plating is likely to save nearly 30 percent in labor costs and will be the least labor intensive process.

TABLE 5. Process Flow Chart (Labor in Minutes)^{4(except dry plating)}

Vacuum Cadmium	Electroplated Cadmium	Dry Plating	
Vapor degrease (14)	Vapor degrease (14)	Vapor degrease (14)	
Grit blast (32)	Grit blast (16), alkaline clean, water rinses, and pickle (25)	Grit blast (32)	
Evaporation coat (40)	Electroplate (10), water rinse, dry (7)	Dry plate (10)	
Inspect (6)	Inspect (6), alkaline clean and rinses (10), dip in cadmium plate and rinse (5)	Inspect (6)	
Chromate conversion	Chromate conversion	Chromate conversion	
Coat (4)	Coat (4)	Coat (4)	
Water rinse, dry (6)	Water rinse, dry (6)	Water rinse, dry (6)	
Inspect (4)	Inspect (4)	Inspect (4)	
Total labor (106 minutes)	Total labor (107 minutes)	Total labor (76 minutes)	

Cost Benefit Summary

The dry plating process could save \$1,000 per day per shift in waste treatment costs and reduce labor costs by 30 percent compared to conventional electroplating.

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Incentives for Dry Plating of 50/50 Zinc-Cadmium Alloy

Based on the coefficient of friction data and microscopic observations, lonEdge concluded that the lubricity of dry plated 50/50 zinc-cadmium alloy is competitive with

the electroplated cadmium. Previously reported data indicate the superior corrosion performance of zinc-cadmium alloys in most environments. Such a study for dry plated zinc-cadmium alloys needs to be conducted. However, since corrosion resistance is a basic material property, it is unlikely that it will depend on the type of process used for plating, unless the coating is highly porous and/or contaminated. The dry plating process plates metals of purity superior to electroplating. These facts lead IonEdge to believe that the dry plating of 50/50 zinc-cadmium is a promising alternative to cadmium electroplating, as it has the potential to reduce waste in the fastener and connector plating industry. 50/50 zinc-cadmium alloy will reduce waste during disposal of discarded components. Also, the technology has the potential for setting a new standard for occupational safety.

As a result of the features and advantages of the dry plating process, several federal agencies, including the EPA (through this grant program and the Small Business Innovative Research program), have contributed to this technology. The dry plating project is now in its third year. During this time, a utility patent was filed with the U.S. Patent Office, and a prototype batch plating apparatus is under construction for cadmium, zinc, and zinc-cadmium alloys. Research facilities, expertise, and analytical support have been provided by the Materials Laboratory of the Colorado State University under a collaborative agreement.

At this time, three potential plating industry segments are being considered for the dry plating technology transfer. These are: discrete components, such as fasteners and connectors plated for a broad segment of users; strip-steel plating for the steel industry; and steel wire coating for the construction industry. Although the current effort is concentrated on discrete components and batch plating apparatus, this technology has considerable potential in continuous galvanizing of strip-steel and steel wire. This is feasible because of the high plating rates achievable in dry plating. Two private plating companies have made commitments for collaboration in the current phases of technology development as well as for the next phases of commercialization. The apparatus development is being tailored to the needs of these companies, each company being in different applications areas. Similarly, new customers will be sought for collaboration in other market segments, and new apparatus will be developed according to their needs. The commercial use of this process will begin in mid-1994.

Potential Barriers to the Dry Plating Technology

Dry plating needs to be tested in pilot-line environments to resolve operational difficulties that new technologies encounter. Some products may prove to be more profitable than others. Like electroplating, the same equipment may not be suitable for barrel plating and rack plating. For example, continuous plating of wire or sheets will require a different configuration of the dry plating apparatus.

One solution to these problems is to set up dry plating equipment within an existing electroplating line, with the plating tank paralleled by a dry plating apparatus. Some products can then be transferred to dry plating over a period of time. Economics and customer satisfaction can be resolved during this period.

The many challenges described here have been resolved for electroplating over many decades. Dry plating is not likely to solve such difficulties in short term, as well. However, as compared to electroplating, dry plating is a relatively lower risk, high payoff, cutting edge technology that can benefit those who are seeking alternatives to conventional technologies.

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PARTICULATE AND HYDROCARBON EMISSIONS REDUCTION DURING WOOD VENEER DRYING OPERATIONS

by

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ABSTRACT

Production Machinery, Inc.'s project assessed if particulate emissions are reduced or eliminated when wood veneer is dried using Radio Frequency (RF) energy as compared to conventional energy sources. Current dryers use natural gas, wood waste, or electric resistance heating in the direct or indirect heating of veneers. The premise is that RF energy, attracted directly to the water in the wood, is able to heat the water and drive it from the wood cells at lower temperatures than are required by conventional dryers. Due to lower temperatures, there is little or no release of hydrocarbons (smoke) from the wood compared to emissions generated from other types of dryers.

INTRODUCTION

PROJECT DESCRIPTION

Outline of Process

Production Machinery, Inc. investigated the particulate and hydrocarbon emission rates from a wood veneer dryer that used Radio Frequency (RF) energy to dry veneer. The emissions, particulates, and hydrocarbons in the form of condensible organics are a result of the veneer being subjected to process handling and elevated temperatures. Sliced wood veneer must be dried prior to its use in products such as overlays, laminates, plywood, and laminated veneer lumber. This drying is required due to the moisture that is introduced during the pre-conditioning process. This conditioning entails exposing the original flitches to either steam or hot water to elevate the temperature and moisture content of the wood prior to slicing. Industry experience has determined that a core temperature of approximately 140°F and a moisture content of at least 40 percent will yield the highest quality slice.

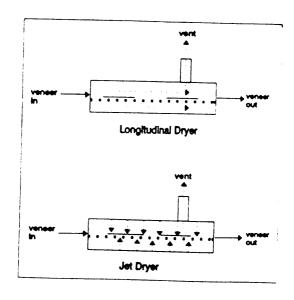
Conventional veneer dryers use hot air blown over individual sheets of veneer. Heating occurs from the outside to the inside to dry the wood. The heat energy is commonly provided by steam heat exchangers (generated with wood hogged fuel) or are direct fired with wood hogged fuel or natural gas. The RF dryer provides internal

heating of the veneer combined with hot air to evaporate vacating moisture from the veneer. This combination of heating methods should promote more efficient drying practices. To establish the benefits of RF drying of veneer, an RF-type dryer (covered by U.S. patent 5,162,629, November 10, 1992) was compared to a natural gas-fired, conventional drying system located on the same plant site.

Unique Process Advantages

Although RF heating systems are commonly used in many industrial applications, its use for drying wood products has been very limited. RF drying of wood products has several advantages over other heating processes used in the forest products industry. Conventional veneer dryers use hot air blown over or at the surface of the veneer, heating from the outside to the inside to provide the necessary heat to all of the veneer for drying. When the frequency of the RF system is properly selected, the majority of the RF radiated energy is absorbed by the water molecules retained in the wood. This energy vaporizes the water, which escapes through the surface of the veneer. As the water concentration decreases, proportionately less RF energy is absorbed in the dryer areas. This selective energy deposition retards the excessive drying of areas in the veneer that are reasonably dry yet allowing continues heating and drying of localized wet spots. In conventional drying systems (see Figure 1), where heat can be provided by either hot air blown across the sheet -- a longitudinal dryer -- or hot air blown perpendicular to the sheet -- a jet dryer -- the heat is conducted through the surface of the veneer for drying. As some areas dry, they still continue to be heated. Conventional veneer dryers operate in temperature ranges from 340° to over 400°F, with recent practices at the lower temperatures to reduce emissions. (This new practice results in lower production rates.) Present methods of drying, because of high temperatures, long drying times, and heating method lead to over-drying or scorching of some areas of the veneer. To reduce scorching, some of the veneer is typically not fully dried. This requires an effort to sort out wet material and return it to the dryer. This wet veneer is referred to as "redry."

Figure 1. Diagram of Conventional Veneer Dryers



In an RF dryer, the drying methods are combined: the RF heats the internal water in the veneer and the blown (forced) hot air reduces the surface layer insulating the wood, thus providing heat to aid evaporation of water from the surface. With the RF dryer, the particulate and hydrocarbon emissions should be reduced due to lower operating temperatures and reduced exposure times to elevated temperatures. In addition, a much higher percentage of veneer should be properly dried in a single RF dryer cycle, that the variance in moisture content within veneer slices should be smaller, and damage to veneers should be reduced due to minimized handling.

Process Schematic

The RF system combines a conventional jet dryer -- where hot air is directed at the surface of the veneer through air caps -- with an RF generator that transmits its energy through a series of electrodes through which single sheets of veneer are conveyed. (See Figures 2 and 3.) This RF configuration is referred to as a stray field system. The remainder of the RF dryer system consists of the support, drive, and idler rolls for transporting the sheets of veneer through the dryer.

Figure 2. RF Generator

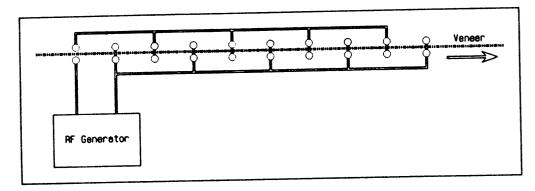
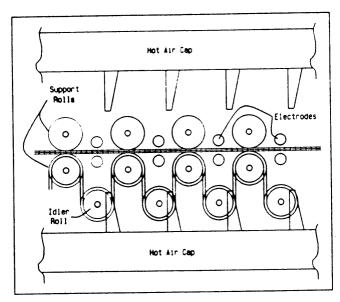


Figure 3. RF Dryer



APPLICATION

Process Replaced

During the drying of wood, water vapor, wood particulates, and volatile compounds are released into the atmosphere. In typical commercial wood veneer drying operations, 20 percent to more than 70 percent of the final dry wood weight will be evaporated as water. The particulate material is wood dust that is eroded from the surface of the veneer during handling. A variety of low molecular weight organic compounds are also released along with the water vapor. The composition and amount of this vapor depends on the species of wood being dried and on the drying schedule or time-temperature history used in the drying process. Low-temperature drying (air drying) of many species is possible, which results in low release rates for these organic compounds. This drying method is of little use in commercial operations because of the long drying times and poor process control. Elevated temperatures are used in forced hot air drying methods to speed water removal rates; however, this results in disproportionate release rates of volatile organic compounds. In most veneer drying systems, heat is passed directly across or at the veneer causing high surface temperatures ranging from 340° to over 400°F. These temperatures are high enough to pyrolize the organic vapors and the entrained wood particulates that are transported out of the dryer's exhaust system. This partially combusted material not only results in the characteristic "blue haze" seen around veneer dryers but also generated highly combustable condensates that coat the inside of the dryer and the exhaust ductwork.

Emissions Reduced

The use of RF heating to dry veneer is expected to improve several process considerations. First, particulate emissions should be reduced because of shorter drying times that reduce handling. Second, the production of partially combusted organic compounds should be lower because of reduced veneer temperature and drying time and the more efficient method of heating with combined RF and conventional forced hot air. The RF system should have significantly higher water extraction rates prior to causing degradation of the veneer. Because the RF is able to direct energy to the location of the moisture, overheating of dry areas will be minimized and the quantities of volatile organics, which are measured as condensible organics, will be significantly lowered.

Another processing advantage of the RF system versus conventional dryers is that the final moisture distribution within a sheet of veneer and from sheet to sheet will have lower variance. This results in a lower dryer reject rate (redry rate) for the RF system, and as a result of reduced handling, particulate emissions are reduced. This lowers operating costs, increases productivity, and improves product quality.

Cross Segment Uses

Although this study shows the potential of the RF drying of sliced veneer for reducing emissions, there are other potential uses of this technology in the forest products industry. The technology could be adapted for drying rotary peeled veneer that is used for making plywood, laminated veneer lumber, and overlays. The RF portion of the equipment also could be used to preheat wood particles and wood

strands -- used in making composite panel products -- prior to drying in rotary dryers. The RF used in this manner would reduce both particulate and hydrocarbon emissions. Other applications include preheating composite panel mats prior to hot processing to improve productivity.

PROCEDURE

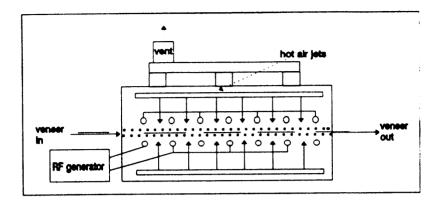
DEMONSTRATION PROCEDURE

Four test runs were conducted, two with the RF dryer and two with the conventional gas fired dryer, of one to two hours per run. Each system, the conventional and the RF, used essentially the same data gathering methods and materials. Two wood veneer feedstocks were used in this study: one stock was sliced, clear, vertical-grain Ponderosa Pine in strips with dimensions of $0.0833 \times 5.00 \times 84.25$ inches; the second stock was rotary peeled Douglas-fir veneer with dimensions of $0.10 \times 51.0 \times 112.5$ inches for the RF dryer and $0.10 \times 27.0 \times 112.5$ inches for the conventional gas dryer. In addition to particulates and hydrocarbon emissions measurements, other process parameters included exhaust conditions, dryer conditions, veneer production, veneer characteristics, and amount of moisture removed from veneer.

The drying equipment used in this study is located at Western Veneer and Slicing, a forest products production facility White City, Oregon. This city is in the Rogue River Valley, which is currently classified by the U.S. Environmental Protection Agency (EPA) as a non-attainment air shed. Two separate production lines were used to reduce the moisture content of high quality sliced veneer from a wet moisture content of approximately 40 percent (oven-dry wood basis) to a dry moisture content of approximately 10 to 15 percent. Both production lines typically use similar feedstocks and are physically located inside a common building.

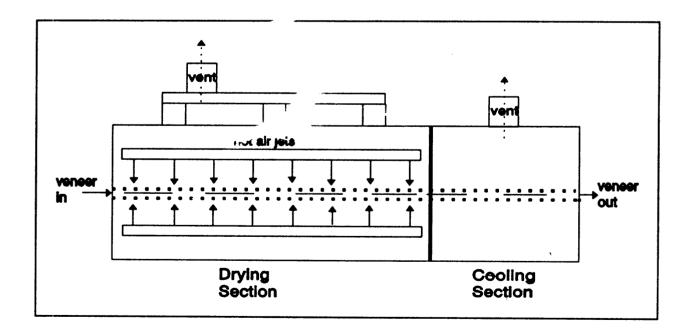
The RF dryer consists of a single-pass conveyer with an active drying length of approximately 24 feet (see Figure 4). The usable conveyer width is 60 inches. This unit used a single RF generator with a maximum rated output of 150 kilowatts operating at a nominal frequency of 3.8 megaHertz. This heating source is supplemented by three natural gas forced draft burners each supplying a maximum of 300,000 BTUs per hour. This unit used a single zone thermostat controller that was set at a nominal temperature of 340°F for the study. The conveyer can be set so that the minimum residence time for veneer in the dryer is 42 seconds. In production, residence times are operator adjusted so that the targeted exit moisture content can be achieved. Residence time ranges from 50 to 90 seconds for the 0.08-inch thick Ponderosa Pine veneer. The system has a single exhaust fan that pulls air through the conveyer's entrance and exit ports and vents via sheet metal ductwork through the roof of the main building. This fan circulates approximately 1,500 ambient cubic feet of air per minute to the environment.

Figure 4. Diagram of RF Dryer System



The conventionally heated veneer dryer is a single pass unit with an active drying length of 58 feet with a usable width of 40 inches (see Figure 5). This is followed by a 13 foot forced air cool down zone. Heat is supplied by 4 forced air natural gas burners each having a maximum output of 300,000 BTUs per hour. The system has a single exhaust fan and vent system that exhausts through the ceiling of the main building. This system circulates approximately 1,150 ambient cubic feet of air per minute. The residence time to dry typical sliced Ponderosa Pine veneer (0.080-inch thickness) is from 80 to 150 seconds. This unit uses a three zone heat control system that is set by plant personnel for this study at an average dryer temperature of 340°F for the three zones.

Figure 5. Diagram of Conventional Gas Dryer System



EVALUATION PARAMETERS

The measured parameters for the study are categorized and defined in the following sections. The experimental methods apply to both the RF dryer and to the conventional natural gas fired dryer. Any differing methods are explained in the appropriate section. To ensure accuracy, a majority of the data were recorded on a Campbell CR21X data logger and transferred to a personal computer for analysis.

Vent Stack Emissions

The test method used for collecting the particulate and hydrocarbon emissions, as well as the flow, temperature, and humidity of the exhaust, is defined in the Oregon Department of Environmental Quality's (ODEQ) book of Industrial Regulations Method 7. An independent testing laboratory, BWR Associates of Medford, Oregon, a company experienced with emissions studies and ODEQ Method 7, collected all vent stack emissions in cooperation with Oregon State University personnel.

Natural Gas Use

Natural gas flow rate was measured using an American Meter Company Model A1-1000 pulse natural gas meter that was installed at each dryer to record total gas use. These units contain a totalizer and a pulsed switch closure that is counted by the CR21X data logger. In addition, a switch closure device was placed on the burner gas solenoids that recorded when the burners were on. One burner was monitored on the RF dryer since all burners were controlled by a single thermostat. On the gas dryer, two burners were monitored since they were independently controlled.

The composition of the natural gas determines the energy given off upon combustion per a specified volume. This value is critical in the determination of energy use. A value of 1,027 BTUs per cubic foot was obtained from the local gas company in reference to Western Veneer and Slicing's natural gas. The gas composition was estimated to be 75 percent methane and 25 percent butane.

The composites of combusted gas, which include water, are critical in that they are part of the vent stack emissions. The amount of water contributed to the system by natural gas combustion was accounted for so that it would not be included as water extracted from the wood. No compensation was made for carbon dioxide or carbon monoxide during combustion.

Exterior/Interior Dryer Ambient Conditions

Wet bulb and dry bulb ambient temperatures were determined for the exterior ambient conditions with an Environmental Tectonics Corporation Psychro-Dial model CP-147 psychrometer at least once during each run. The interior temperatures were taken from thermocouples that were permanently mounted in the dryers. The temperature and specific moisture content values of the air exhausting from the dryer were obtained from BWR Associates' test results.

Material Physical Parameters

The main veneer parameters were the temperatures before and after the dryer, the weights before and after the dryer, and the moisture meter reading after the dryer. The targeted moisture content for material exiting the dryer was 10 percent (oven-dry wood basis) for the sliced veneer and 5 percent for the rotary peeled veneer. Of the total material going through the dryer, 5 to 8 percent of the total number of veneers dried was sampled. From this fraction, the moisture content and weights in and out of the dryer were recorded. Sampling 5 to 8 percent of the material yields a representative sample size. Individual samples within the fractional sample consisted of a group of 4 veneer slices that were end-marked prior to the dryer to ensure that they could be identified when exiting the dryer.

Veneer moisture content measurements were taken after drying. Green end measurements were not taken because the veneer was above fiber saturation point and were off-scale on the moisture meters. On-line moisture contents were measured with a hand-held, dielectric moisture meter (Ward Systems, Inc. Model 510) and recorded. A 1-inch thick piece of styrofoam was placed under each sliced veneer pair to isolate the reading. Since this type of meter is dependent on wood thickness, two combined slices of the sliced veneer (a double thickness) were needed to obtain a correct reading. The recorded moisture content for the sliced veneer was an average of 6 readings within each sample group of 4 slices (3 readings from 2 veneers and 3 from the other 2 combined veneers). To ensure that the moisture meter was reading correctly, 7 to 8 samples (groups of 4) evenly spaced throughout the run were measured with the moisture meter and weighed. These were taken to the laboratory to be oven dried to determine their actual moisture content. A regression equation was developed that gave the true moisture content as a function of the moisture meter reading. Moisture content out of the dryer for the rotary peeled veneer was monitored by the same hand-held meter as the sliced veneer, but the values were not recorded.

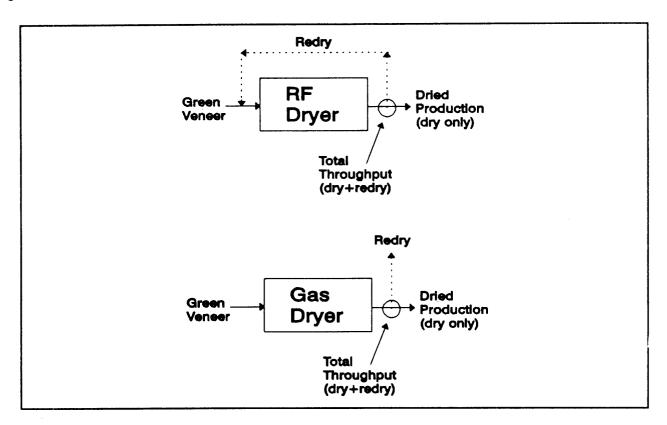
The sliced veneer's ambient temperature before the dryer was defined to be the same as the conditioning hot water bath temperature of 150°F. The temperature of the rotary veneer before the dryer was at ambient temperature since it had been stored in the plant for 1 or 2 days in a bundled unit at room conditions. Since the dryer conditions remained fairly constant, only a small number of temperature measurements were made on individual pieces after each dryer run using a Scotch infrared temperature sensor.

The samples of sliced veneer, from which moisture meter readings were taken (the 5 to 8 percent of total throughput), were also weighed before being run through the dryer using an OHAUS GT4100 scale and immediately after the dryer with an OHAUS I-10 digital scale. Due to the length of the veneer, a support $(1.5 \times 3.5 \times 4.0)$ feet of pine) was placed on the scale and its weight tared out before the veneer was weighed. The exit moisture content, weight loss, and number of sheets dried were used to determine the moisture loss for the sliced veneer. Moisture loss for the rotary peeled veneer was determined by weighing the total amount of veneer before and after drying using a truck scale. The difference was the total water loss from drying.

The total number of slices passing through the dryer during the run was recorded. Redry and fully dried material were tallied. Figure 6 illustrates how veneer tallies were made in terms the types of veneer production through the dryer as total

dryer throughput and total dry production. This tally was done at the output end of the dryer when the run was over. The EPA uses the total throughput basis for its calculations and considers the redry rate. However, PMI felt that a truer measure of a plant's production would be the total dry basis, so both basis were used. On the RF dryer, the redry material was sent back through the system, so a running tally was kept. On the conventional dryer, the redry material was kept separate and not run back through the system during the run.

Figure 6. Flow Diagram of How Veneer Tallies Were Made



The length, width, and thickness were recorded from the veneer samples brought back to the laboratory to be oven dried (48 to 52 pieces from each run). Their length and width were measured with a Stanley 25-foot tape measure. The thickness was determined with a Starrett hand-held micrometer, accuracy to 0.01 millimeter. The number of sliced and rotary peeled veneer sheets dried were conducted at the end of each run. No distinction was made between dry and redry for rotary peeled veneer. All material passing through the dryer was considered to be fully dried material.

The criteria for wood quality included a visual inspection of the same 5 to 8 percent that was sampled for weight and moisture content. The visual inspection involved discoloration and appearance of cracks or other drying related defects apparent in the wood.

ASSESSMENT SUMMARY

While the project showed that the RF dryer could reduce emissions for sliced veneer -- the type of veneer the equipment was designed to dry -- care must be taken in using the results of this study beyond its scope since only one trial run was made. Although the RF dryer was able to dry the rotary veneer, it could not do so in an efficient manner during the test. The RF dryer was calibrated specifically for sliced veneer, and it is expected that a recalibration of the dryer for the rotary peeled veneer would improve the overall performance.

COST OF DEMONSTRATION

The total cost of the project was \$27,727.87. EPA contributed \$21,652.87; PMI contributed \$1,075; and Pacific Power and Light donated \$5,000 towards the study.

RESULTS AND DISCUSSION

PERFORMANCE RESULTS

The RF dryer reduced particulate emissions by 40 percent and condensible organics by 23 percent. Condensible inorganics (that occurred at near background levels) were not reduced for the sliced veneer drying when considering total dried production (see Table 1). When the veneer tally is based on total veneer throughput, the emissions reduction is less, 18 percent for particulate and 0 percent for both the organic and inorganic. The particulate component on a weight basis represents approximately 84 percent of the total emissions measured.

TABLE 1. Summary of Emissions Reduction for RF Drying of Sliced Veneer

Emission Component	Reduction in Emissions (%) Based on Total Dryer Throughput	Reduction in Emissions (%) Based on Total Dried Veneer
Particulate	18	40
Organic	0	23
Inorganic	0	0
Total Emissions	16	38

The run of rotary veneer through the RF dryer was unsuccessful in reducing emissions. The equipment was able to dry the veneer, but because the "tuning" of the RF generator was specifically tailored to the sliced veneer, the RF generator would not load properly, providing only about 25 percent of its expected output power. If the generator were tuned to full size sheets of veneer, it is expected that the RF dryer would also reduce emissions for the rotary veneer as it did for the sliced veneer.

PRODUCT QUALITY VARIANCE

The RF dried sliced veneer was "brighter and less brittle" than the veneer dried in the conventional dryer, thus better maintaining the quality of the veneer. This observation could not be quantified, but was made by the experienced veneer graders who were working on the production line at Western Veneer and Slicing.

CONDITIONS THAT IMPACT PERFORMANCE

A number of production parameters affect the operating efficiency of the RF dryer, and only those conditions reported in this study were evaluated. Such conditions that impact dryer performance are: material, environmental, and equipment parameters. Material parameters are percentage of energy contribution by RF and conventional heating, tuning of the RF, exhaust flow, temperature setting, and residence time. Environmental parameters include temperature and humidity. Although not studied, equipment could be redesigned once the specific operating conditions are defined to provide similar, if not greater reductions in emissions when drying veneer. For example, the performance of the RF dryer could be enhanced further by reducing the amount of exhaust flow. This would improve energy efficiency and reduce natural gas combustion, which in turn would reduce hydrocarbon and water vapor emissions.

TABULATION OF DATA

Comparison of emissions between the RF and conventional dryers was performed under the following parameters: particulate emission; condensible organic and inorganic emission; and water vapor emission.

In order to place both drying systems on an equal material basis, two sets of computations were performed. These results present the emission rates on two different basis: (1) emission rates relative to the total volume of material exiting from the dryer -- total throughput of veneer; and (2) emission rates relative to the volume of finished dry veneer product -- total dry production. The EPA uses the total throughput basis at a given redry rate; if the rate changes, the dryer needs to be recertified. Both basis provide useful information about dryer efficiency, and PMI has provided data in the two common forms seen in the literature. The emission rates relative to the total amount of dried production for the full production cycle appears to be the more relevant. The two sets of emission data are provided in Tables 2 and 3.

TABLE 2. Summary of Particulate amd Hydrocarbon Emission Rates [(pounds/(hour * MSF 3/8 inch basis)]

Emission Component	Run 1 RF Dryer with Sliced Veneer		Run 2 RF Dryer with Rotary Veneer	Conv Dryer v	un 3 entional vith Sliced eneer	Run 4 Conventional Dryer with Rotary Veneer
	(A)	(B)	(A)	(A)	(B)	(A)
Particulate	0.124	0.135	0.189	0.152	0.226	0.079

Condensible Organic	0.021	0.023	0.050	0.020	0.030	0.022
Condensible Inorganic	0.002	0.002	0.006	0.002	0.003	0.003
Total Emissions (1)	0.147	0.160	0.245	0.174	0.259	0.104
Total Water Vapor (2)	179	196	408	195	290	214

MSF: 1,000 square feet (surface measure) of veneer at a nominal thickness of 3/8 inch; 1 MSF of veneer equals 31.25 cubic feet of wood

(A) Based on total veneer through dryer(B) Based on total dry veneer through dryer (total-redry)

(1) Particulate + Condensible organic + Condensible inorganic

(2) Wood extract + Combustion products + Make up air

TABLE 3. Specific Release Rates (pounds/MSF)

Release Rate	RF Di	un 1 ryer with I Veneer	Run 2 RF Dryer with Rotary Veneer	Run 3 Conventional Dryer with Sliced Veneer		Run 4 Conventional Dryer with Rotary Veneer	
	(A)	(B)		(A)	(B)		
Particulate	0.178	0.195	0.299	0.212	0.316	0.111	
Condensible Organic	0.031	0.033	0.079	0.028	0.042	0.030	
Condensible Inorganic	0.003	0.003	0.010	0.003	0.005	0.003	
Total	0.209	0.241	0.388	0.243	0.363	0.144	
Water Vapor	258	282	408	273	406	299	

(A) Based on total veneer through dryer(B) Based on total dry veneer through dryer (total-redry)

• Émission rates are pounds per hour

• All production runs are assumed to be done at typical production throughput rates as given in Table 1

As can be seen in Table 2, the particulates release rates for sliced veneers have a higher level for the conventional dryer than for the RF dryer (run 1 versus run 3) for both tally basis computations. As the feedstocks for both runs were identical, no material differences should have caused this difference. The RF particulate emission rates are 18 and 40 percent lower, based on total throughput and dried production respectively, than the conventional dryer. These values are well above the experimental uncertainty. A significant reduction of 23 percent exists for condensible organics (hydrocarbons) on the total dried production basis, no difference occurred on a total throughput basis. Release rates for inorganic material trapped in the condensible organic fraction is a very small part of the total release for all runs. These values are near the minimum measurable levels for this test.

For the rotary peeled veneer (run 2) dried with the RF drier, the RF system failed to load properly and is therefore not representative of what this technology can provide. This run is an example of a poorly implemented conventional veneer dryer, since the output of the RF was much lower than its rated capacity, resulting in a larger contribution of natural gas heating. Excessive local heating was observed near knots. Some knots were severely discolored or dislodged during the drying process. A change in dwell from 6 to 4 minutes during the run eliminated much of this problem. As a result, significant amounts of wood debris were generated and entrained in the circulation air and ejected from the system. Run 2 also produced approximately a factor of 2 higher levels of condensible organic release than the other 3 runs. This is consistent with the harsh heating observed near knots.

The release of water vapor from the dryer was also assessed. Although water vapor is not considered an environmentally sensitive material, it is what most casual observers see emanating from veneer dryers. These data show a significantly elevated release from Run 2 as compared to the other 3 runs. This is due largely to the very low production rate for this trial. The longer the veneer residence time in the system, the larger the water vapor contribution made by the natural gas burners and the makeup air stream relative to the amount of water contributed by the wood. Water vapor generated by the burners plus the water vapor present in the makeup circulation air stream contribute 25 to 40 percent of the total emitted water vapor in these trials. If one computes water emission rates assuming that all 4 runs were done at the maximum throughput rates for the material being dried and then compensate for the water vapor added by natural gas combustion and external makeup air for Runs 1 through 4 respectively is 7.08, 7.15, 7.63, and 7.33 pounds per square foot.

The particulate emissions rate for rotary peeled veneer should be measurably higher than the sliced veneer because of the dramatic differences in surface texture between the two veneering operations. Data presented in Tables 2 and 3 for Runs 3 and 4 indicate that the particulate emissions for the sliced veneer were between a factor of two to three times higher than the rotary peeled veneer. Possibly, the large fragments seen with the rotary veneer do not get entrained in the exhaust air of the conventional gas fired dryer and thus do not appear in the test samples. This may also be influenced by the width of the veneer relative to the design width of the dryer's conveyer system that would hamper vertical air flow. Although the exhaust rates for both dryers are similar, the internal volume of the conventional dryer is much larger than the RF system, which may play a role in the entrainment of particulates in the exhaust air stream.

COMPARISON OF STUDY DRYERS TO OTHER COMMERCIAL DRYERS

The emissions from these two veneer dryers can be compared with those from other installations. Information provided by the ODEQ on veneer dryers for two softwood plywood plants appear in Table 4. These data are for installations that use rotary peeled Douglas Fir sheets similar to those dried in this study. No particulate emission data have been found for sliced veneer. Volatile releases from sliced and rotary peeled veneer are assumed to be the same and depend on the wood species, temperature, and dwell. Ponderosa pine, because of its chemistry, would be expected to have higher values of organic emissions. For the Site 1a data, the dryer was out of compliance for opacity (smoke) and required production adjustments. Site 1b data are more representative of a compliant system. For the compliant tests the particulate

emission levels ranged from 0.09 to 0.18 pounds/(hour*MSF 3/8 inch) basis. Condensible organic rates for PMI's tests fall in the same range as the results of the ODEQ test data.

TABLE 4. Summary of Particulate and Hydrocarbon Emission from Other Veneer Drying Operataion Compared to thi Study on a Total Throughput Basis

Emission Component	Site 1a	Site 1b	Site 2	Run 1	Run 3	Run 4
	non compliant	compliant	compliant	compliant		
Particulate	0.827	0.0915	0.177	0.124	0.152	0.079
Condensible organic	0.168	0.0249	0.0128	0.021	0.020	0.022
Condensible inorganic	0.0196	0.0018	0.0026	0.002	0.002	0.003
Total (2)	1.01	0.118	0.192	0.147	0.174	0.104
Water vapor (3)	790	449	329	179	195	214
Wood species	Douglas fir sapwood	Douglas fir sapwood	Douglas fir sapwood	Ponderos a pine	Ponderos a pine	Douglas fir

(1) Emission rates are: lbs/(hr* MSF 3/8 inch) basis

(2) All production runs are assumed to be done at typical production throughput rates
(3) Particulate + Condensible organics + Condensible inorganics
(4) Wood extract + Combustion products + Makeup air
(5) Site 1a: 8.81 MSF/hour, test run #1, 0.10 inch thick sap; this run was smoking heavily and was out of ODEQ levels

(7) Site #2: 5.689 MSF/hour, 0.10 inch thick

The ODEQ standard for opacity was not done during the project. Although not documented in the literature whether there is a relationship between the emission concentrations measured as particulate and condensibles and the opacity measurements, PMI suspects there is a positive correlation between the product of concentration, the highest dryer internal temperature, and the amount of time the particulates and organics remain at high temperature. The lower residence time, coupled with lower peak temperatures of the veneer in the RF dryer, may lead to significantly reduced smoke generation. Secondary evidence to support this idea can been seen in the significantly reduced internal coatings of extractives in the RF dryer compared to the conventional dryer.

COST/BENEFIT ANALYSIS

Higher production efficiency can be realized when using RF technology in veneer drying. For example, the RF dryer processed 636 square feet of veneer per

⁽⁶⁾ Site 1b: 5.362 MSF/hour, test runs #2 and #3, 0.228 inch thick; these runs were within permitted levels after adjustment from Run #1

hour more than the conventional dryer during this test (based on total dry production). Assuming approximately \$0.50 per square foot sale value, the RF dryer was producing \$318.00 of veneer per hour in excess of the conventional dryer. This amounts to over \$600,000 in additional sales annually.

The RF project illustrated a number of benefits to industry. Some are difficult to quantify, as they pertain to variables such as the environment, or they are based on matters of opinion, such as aesthetic veneer quality. RF drying seems to be more efficient compared to conventional drying. The natural gas savings for the RF dryer obviously is offset by electrical consumption, but considering actual dry veneer production, the RF dryer consumed approximately 38 BTUs per hour per square foot of veneer less than the conventional dryer in this test. The conventional dryer also generated approximately 23 percent more redry material than the RF dryer, which equates to more run time and energy usage.

The RF dryer requires only about half the space of an equivalent conventional machine. This allows the user to further optimize utilization of the floor space required for plant operation. Due to cleaner emissions, the maintenance requirements for the RF dryer are far less demanding than its conventional counterpart primarily because of the reduced levels of pitch and resin buildup within the dryer.

RF drying also proves to be advantageous when evaluating the residual moisture distribution after the drying process is complete. Wet spots can be a problem for a conventional dryer, whereas in RF drying, a very uniform, normalized product is produced. RF energy is attracted to any contained moisture within the veneer and works only on those areas, without affecting zones that are already dry.

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Incentives

RF drying technology has a great deal to offer in today's wood products industry marketplace. Considering natural resource shortages and environmental issues affecting it's producers, the benefits associated with dielectric heating will have an impact. In an attempt to do more with less, there has been a major trend towards the production of thinner and thinner veneer products. As mentioned previously, all of this veneer will have to be dried as part of the process. RF drying will provide one of the safest and efficient methods of handling this valuable resource with the least amount of impact on the environment. RF drying methods can aid manufacturers in keeping up with the demand for veneer while remaining competitive and can eliminate cost overruns created by excessive redry runtime and compromised veneer quality.

Phase of Development

The Dry-Tech Radio Frequency Veneer Dryer, U.S. Patent #5,162,629, is currently in production status. To date, two machines have been built, shipped, and installed for the same customer in White City, Oregon. PMI is responding to considerable interest in the RF dryer, including a number of international inquiries.

Further development is being considered to modify this technique for use in the plywood industry. Redry and wet spots are ongoing problems for plywood applications that could be rectified through the use of RF drying.

Target Industry Potential

This technology could be easily adapted for drying of rotary peeled veneer, which is used for making plywood, laminated veneer lumber, and overlays. The RF portion of the equipment could also be used to preheat wood particles and wood strands -- used in making composite panel products -- prior to their being dried in rotary dryers. Other applications include preheating composite panel mats for hardboard, particleboard, medium density fiberboard, and oriented strand board prior to hot pressing to improve productivity.

CONDUCTIVE POLYMER COMPOSITES TO REPLACE HEAVY METALS IN COATINGS AND ADHESIVES

by

Harry S. Katz and Radha Agarwal Utility Development Corporation Livingston, NJ 07039

ABSTRACT

Improved polymer matrix conductive coatings and adhesives are needed to replace current products that use heavy metals. Utility Development Corporation developed a low-cost polymer adhesive that has equivalent conductivity to those currently available and assessed its physical and electrical properties. The project focused on the inclusion of short graphite fibers, carbon/graphite microspheres, and conductive carbon powder fillers as the conductive elements.

INTRODUCTION

PROJECT DESCRIPTION

Utility Development Corporation developed and produced small quantities of conductive polymer composites as substitutes for heavy metal solders and other metallic components, such as lead, tin/lead, and silver, in electronic applications. The main formulation contains carbon/graphite fibers and carbon/graphite fillers in a thermoplastic matrix. Most formulations were extruded in a one-eighth inch diameter rod that could be used in the same manner as conventional solder.

In addition, Utility Development formulated and tested a number of conductive polymer adhesives and coatings. In these studies, the carbon/graphite fibers and fillers were dispersed in water-based urethane and in epoxy resins.

The project focused on the formulation and experimental testing of the conductive adhesives. As Utility Development has limited experience in the electronics field, this program did not accent specific end uses.

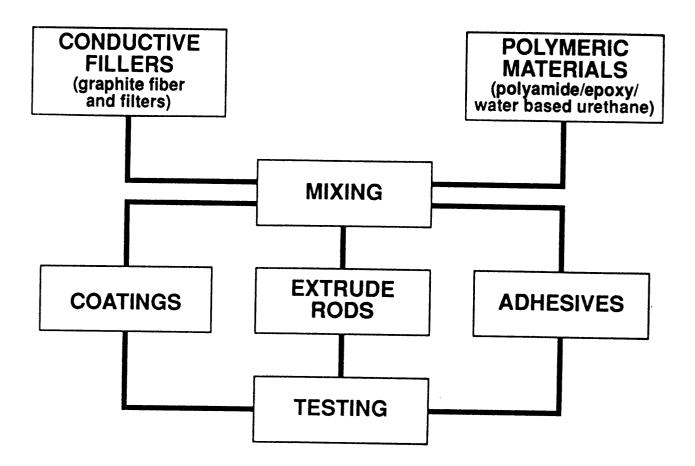
Unique Product Features/Advantages

The low-cost and environmentally safe conductive fillers should provide high performance for the many applications usually requiring the use of a lead-based solder. In conventional soldering, flux and solvent are required to obtain a clean surface and good bond strength. The use of polymer matrix conductive composites do not require fluxes or solvents.

Process Schematic

The process schematic for polymer matrix conductive products is shown in Figure 1.

Figure 1. Polymer Matrix Conductive Products



APPLICATION

Process/Products Replaced

Conductive polymer matrix solders, coatings, and adhesives were formulated to replace current products that use heavy metals such as lead, tin/lead, and silver. These metals, which are used extensively in electronic devices and automotive control systems, are potential toxic contaminants to the environment.

Wastes Prevented

The conductive materials replace products containing heavy metals, such as lead, tin/lead, and silver. Fluxes and solvents (e.g., methyl ethyl ketone, toluene) used in a conventional soldering process are also eliminated.

Cross-Segment Uses

Conductive adhesives may be used where a convenient, low temperature-curing conductive material is desired. These conductive composites may be used by the electronics industry to replace lead and tin/lead solders. They also may be used as coatings, antenna, or circuitry on "smart" credit cards and electromagnetic shielding.

PROCEDURE

DEMONSTRATION

Polymer matrix solder materials, conductive adhesives, and coatings were produced by using a high loading of conductive fillers in various polymer matrices. Most of the work was conducted with different types of carbon/graphite fibers, flakes, and powders at various loading levels in order to determine the proper materials and formulation for a highly conductive polymer composite.

Utility Development assessed three grades of Union Camp's polyamide resin: EPI REZ 2641-D, EPI REZ 2643-D, and EPI REZ 2645-D for use as the matrix of the conductive composites. The melting point of these resins ranged from 120° to 140° centigrade.

A two-component thermoset epoxy resin system was also evaluated. This system consisted of Shell Epon 826 with Henkel Versamid 140 as the curative. Both components were highly loaded with conductive fillers before mixing and curing.

Conductive fillers that were evaluated included ten different grades of graphite powders and a special grade of very high electrically conductive graphite filaments. Representative products included P-120 Graphite Filaments (AMOCO Performance Products), CABOT-XC72R Graphite Powder, and M201S Milled Graphite Fiber (Kreha Corporation). The filaments were chopped or broken during mixing procedures to obtain a very low aspect ratio.

A mini-mixer injection-molder quickly mixed and molded many different permutations of fillers. The temperature and mixing cycle were varied for the different formulations. The resultant product was a continuous extruded rod, approximately one-eighth inch in diameter, and looked like standard commercial solder, except for the black color.

In the beginning of the project, the conductivity levels of the formulations containing P-120 were not consistent. This graphite fiber is sold in continuous multifilament spools. In early experiments, filaments were broken into irregular lengths using the Mini-Max Molder to obtain a low aspect ratio for the flexible rod-type solder product. Since one factor for erratic conductivity levels could be attributed to the uncontrolled aspect ratio of the graphite fibers, Utility Development elected to ball-mill and screen-separate the filaments, selecting a narrow length range. After the P-120 fiber was ball-milled, the broken fragments were screened through 50, 100, and 200 mesh screens. +50 and -200 mesh fibers were discarded, and -50 and +100 mesh fibers were used. More consistent conductivity measurements were obtained using the ball-milled fibers.

To achieve the maximum conductivity, very high loadings of conductive fillers are required in the polymer (one part resin to at least two parts filler by weight). At these high levels of fillers, the adhesion to the substrates becomes very poor. Utility Development evaluated different resins and coupling agents, such as silanes and titanates, to improve adhesion, but were not initially successful. After the preliminary fiber screening tests, Utility Development investigated proper filler packing concepts and selected the best combinations and ratios of fillers and fibers. This minimized the problems of poor adhesion to the substrates. Various ratios were tested to optimize the combinations in accordance with packing concepts to achieve good adhesion, maximum packing, and high electrical conductivity.

Packing Concepts

Packing concepts are vital to the success of the polymer composite. All conventional polymers are good electrical insulators. In order to obtain a polymer composite/adhesive with extremely high electrical conductivity, filler loading in the polymer must be maximized. If packing concepts are not used, a very high loading of filler results in a composite with very poor physical properties and a high void content due to processing problems with resin-starved polymer solder.

Examples of the effectiveness of good packing combinations are illustrated in Figures 2 and 3, which are taken from the *Handbook of Reinforcements for Plastics*, published in 1987 by Van Nostrand Reinhold. (Mr. Katz, the principal investigator of this project was co-editor and contributing author of this book.) The co-editor of the Handbook, Dr. John V. Milewski, was the main proponent of the development of packing concepts.

Figure 2. Addition of Small Spheres to Large Spheres

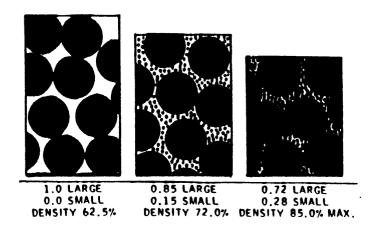
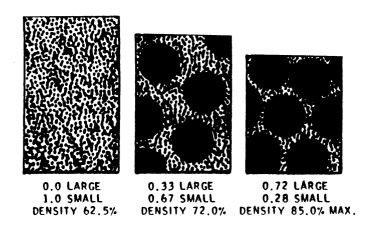


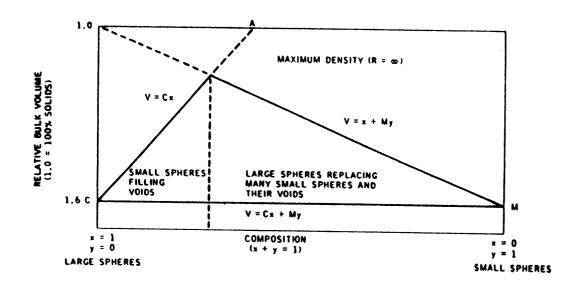
Figure 3. Addition of Large Spheres to Small Spheres



Figures 2 and 3 illustrate the densification that occurs when small spheres are added to large spheres. Maximum density is obtained when the small spheres are packed to their maximum density within the voids of the larger spheres. Each illustration represents the same volume of solid material; thus, the relative bulk volume decreases as densification occurs. In Figure 2, in each step towards greater density, a large sphere is removed and the same amount of solid material is replaced by small spheres within the voids of the remaining larger spheres. Figure 3 illustrates how densification occurs by the opposite process, in which a number of small spheres and their associated voids are removed, and the same amount of material is replaced as one large solid sphere.

The theoretical packing curve for two different size spheres is shown in Figure 4. The solid line is a theoretical packing curve for an infinite size ratio $R = \infty$. For this example, the ratio R is the diameter of the large sphere divided by the diameter of the small spheres. The maximum density point represents the condition illustrated in Figures 2 and 3 at the extreme right. In Figure 4, the composition of the mixture is shown by the horizontal scale X, the volume fraction of the small spheres, for a total volume of unity. The left-hand ordinate is the relative bulk volume and is defined such that 1.0 is equal to 100 percent solid material (100 divided by the percent theoretical density). A material with a relative bulk volume of 1.6 would be 62.5 percent theoretically dense. Thus C gives the experimentally determined packed volume of the large spheres, and M gives that of the small spheres. By using relative bulk volume, rather than percent theoretical density, the packing curves for the infinite size ratio becomes two straight lines.

Figure 4. Theoretical Packing of Two Sphere Systems



A proper ratio of the diameters of two or three sizes of fibers and microspheres results in good interstitial packing and a minimum void content. Under these conditions, there will be a lower resin demand to fill the interstitial voids and coat each filler particle so that adequate flow is maintained during the molding procedure. Also, the molded part will have less air pockets or microvoids that reduce the physical and electrical properties. In contrast, if sphere ratios or particle size distribution is wrong, the result will be poor flow, high void content, high shrinkage, high electrical resistance, and low physical properties.

For conductive composites, coatings, and adhesives, 1 part of resin to 1.5 parts of a combination of fillers was found to be an optimum ratio to obtain good adhesion and conductivity. Also, 3 parts of the ball-milled carbon fiber to 1 part of carbon powder yielded a good packing combination.

EVALUATION PARAMETERS

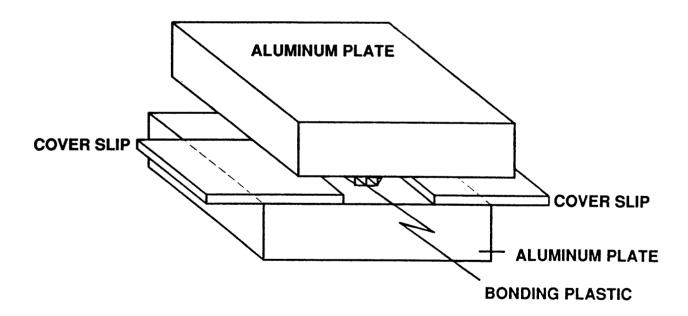
Dr. Haim Grebel, Professor at New Jersey Institute of Technology, supervised conductivity tests at NJIT. The procedures are described below.

Use of Conductive Polymer Composite as Solder for Metals

Aluminum was selected as the metal to be bonded. It was machined into two plates, 1 x 1 x 0.078 inches (25.4 x 25.4 x 2 millimeters). The conductive polymer composite samples containing resin, graphite fibers, and powder were cut into beads, each of equal volume (10.5 cubic millimeters). The samples were in the form of rods, and the volume of the beads was estimated assuming they were perfect cylinders.

The diameter of the beads was measured using a micrometer, and the length of the bead was cut to provide the same volume in each sample. In order to obtain the same thickness of the bonding material between the plates, cover slips, 0.0085 inches thick, were used as shown in Figure 5.

Figure 5. Experimental Configuration for Testing the Soldering of Polymeric Materials



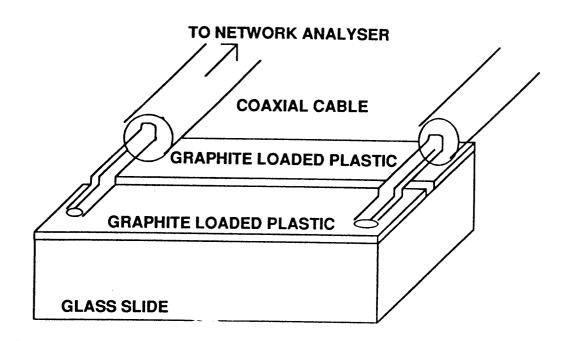
Both the plates and the plastic bead (positioned on one of the plates) were heated on a hot plate to the point where the bead became tacky. The second plate was then placed on the plate holding the bead. This "sandwich" was removed from the hot plate and pressed together for approximately 30 seconds and allowed to cool.

The temperature of the hot plate was monitored using a thermocouple. The cover slips were removed and the resistance bond measured at direct current (dc) and at 1 kilohertz. A control measurement was made using tin/lead solder. The error in resistance values was estimated at 7 percent; this was based on the possible error in the volume of solder bead.

<u>Transmission Characteristics of a Slotline Made of Conductive Polymer Adhesives at Microwave Frequencies</u>

A film was cast on a glass slide of a formulation containing water-based polyurethane and graphite filler. This conductive polymer film was pressed between two heated microscope slides, 2 x 1 x 0.039 inches, to form a thick film. Prior to deposition, the glass slide was cleaned sequentially with acetone, methanol, and distilled water to remove particles that would alter the conductivity of the film. To create a slotline, the film was ablated longitudinally at its center using a high power ultraviolet laser. A pair of coaxial cables were connected to the slotline by pressing the former against the latter, as shown in Figure 6.

Figure 6. Experimental Configuration to Test High Frequency Response of the Filled Polymers



Pieces of low-resistance conductive polymer composite thick film (UDC 24R) were placed between the coaxial line and the slotline for better contact. An HP Lightwave Component Analyzer was used as a network analyzer to determine the Sparameters of the line, and an HP 7475A plotter was used to plot the characteristic curves.

The resistance between two close points on the film was found to be 30 ohm (as measured by a multimeter). The contact resistance between the film and the coaxial line was also found to be 30 ohm. Thicker films had lower resistance, better transmission characteristics, and produced a lower mismatch at the transition points compared to the thinner films (of the order of tens of m that were developed by spinning over glass slides).

COST OF DEMONSTRATION

The cost of this demonstration project was \$59,337. EPA provided \$25,000 through the Pollution Prevention By and For Small Business Grant Program, and Utility Development contributed \$34,337.

RESULTS AND DISCUSSION

PERFORMANCE RESULTS

Results indicate that the conductivity of the plastic materials filled with graphite fibers and powder may be suitable for many soldering applications. These materials need to be tested in actual applications, and additional research may be necessary to refine the product.

PRODUCT QUALITY VARIANCE

Product quality variance depends on such factors as method of application, thickness of the coating or solder joint, temperature, and mixing time and speed of the formulation. All of these factors can be varied within a wide range after first establishing product and process specifications.

CONDITIONS THAT IMPACT PERFORMANCE

The uniformity of dispersion of the conductive fibers and fillers is one key performance factor of the conductive polymer systems. Another condition that must be considered is the presence of moisture in the polymer solders and coatings, either before or after application. One of the primary matrix materials tested was a series of polyamide resins that have excellent adhesion, physical properties, and application characteristics. However, they tend to adsorb moisture. These solders and coatings should be completely dry before conductivity measurements are taken.

TABULATION OF DATA

Conductive Polymer Composite as Solder for Metals

Bulk resistance of the aluminum plates, measured with a multimeter, was 0.2 ohm. Contact resistance between the multimeter probes and the plate measured 0.1 ohm.

The resistance observed at dc and at 1 kHz was practically identical. The actual electrical resistance measurements of various soldering materials are given in Table 1. As can be seen from the Table, some filled polymer solder materials possess relatively low resistance values, especially UDC 24, as compared to the control metal solder.

TABLE 1. Electrical Resistance of Filled Polymer Materials

Filled Polymer Sample Tested	Volume of Sample	Electrical Resistance (ohm) *
UDC 23R	10.5 mm ³	24, 56
UDC 24R	10.5 mm ³	8, 1
UDC 26R	10.5 mm ³	75, 70
UDC 27R	10.5 mm ³	50, 28, 37
UDC 28R	10.5 mm ³	70, 45, 63
UDC 29R	10.5 mm ³	47, 77
Control Metal Solder		,
Pb/Sn: 50/50 by weight		.6
Pb/Sn: 60/40 by weight		.4

Note: Aluminum is a difficult metal to solder and poorly adheres to many other materials including many conductors.

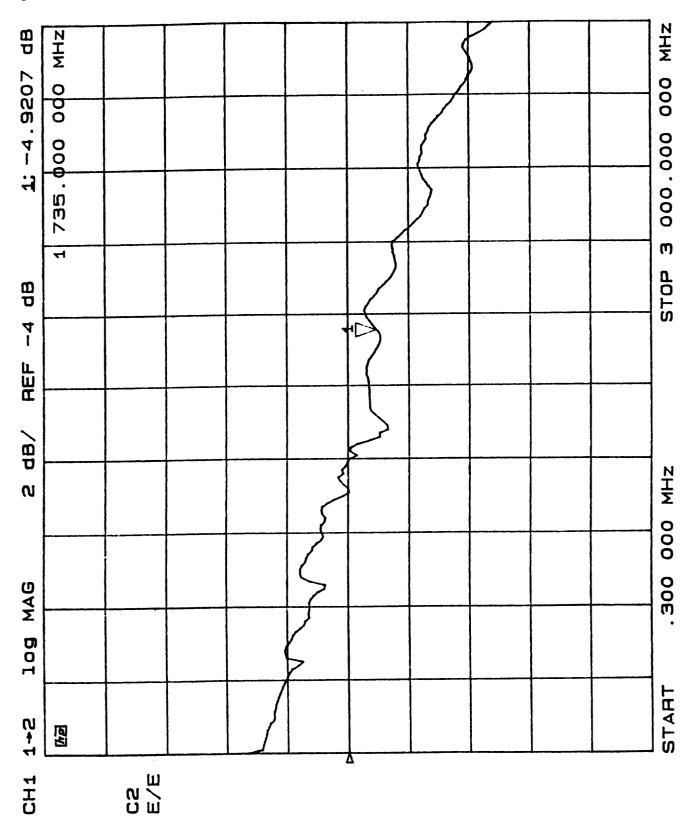
- They exhibit relatively low resistance values that may be tolerated by the electronics industry.
- They are environmentally safe.
- The contact resistance problem can be solved by using other polymer materials or coupling agents. Soldering materials are usually matched to the electronic circuit materials even though both the circuit and the soldering material are good conductors. For example, the electronics industry uses silver alloy as soldering material to match the silver-made circuitry layout.

<u>Transmission Characteristics of a Slotline Made of Conductive Polymer Adhesives at Microwave Frequencies</u>

The S-parameter plots for the slotline may be seen in Figure 7. The transmission coefficient (S21) is greater than -10 decibels for frequencies up to 1 gigahertz (the curves were plotted over the range of 0.3 megahertz to 3 gigahertz).

^{*} The resistance value contains two parts: the resistance of the contact (the interface between the filled polymer and the aluminum plate) and the bulk resistance of the filled polymer itself. Most of the resistance values shown in the Table are attributed to the interface resistance. That was confirmed by using longer strings of polymeric solder material. Utility Development concluded that contact resistance is significant for small volume of polymeric beads. This explains the large variation in resistance values, since difference in adhesion characteristic may result in variations in resistance values. Nevertheless, Utility Development believes that polymeric soldering materials are promising for the following reasons:

Figure 7. Voltage Transmission as a Function of Frequency



Suitable conductive plastics as a substitute for metal components have great potential for use in microwave integrated circuits. The application of these trials on actual circuit boards was not performed.

Utility Development was successful in obtaining relatively low electrical resistance, ranging from 0.2 to 20 ohms, with conductive fibers and fillers in polymer matrix composites. Even though this resistance is not as low, about 0.5 to 1.0 ohm, as the heavy metal solder materials, there should be many applications for this performance level, and future work will provide improvements in further resistance level reductions.

The major advantage of composite polymers is that they are conductive at low and high (up to 1 gigahertz) frequencies. Several devices may now be realized in plastic materials: short connections between wires, plastic antennas, and high frequency circuitry. The dispersion of the carbon/graphite fillers inside the polymeric matrix dictates the low and high conductive properties of the overall material. This can be achieved by using the same polymeric matrix by varying the amount of conductive filler in that matrix. Dr. Grebel has stated that these types of conductive polymer composites will have an advantage for some applications since "conventional lead solder or other conductive metals, such as copper, possess mainly one degree of conductivity which is hard to vary under normal operating conditions." One may postulate that conductive polymers may be made into shields for electromagnetic radiation by varying the amount of conductive filler in the matrix. This is because the transmission drops sharply at very high frequencies as can be seen from Figure 7. The frequency at which the transmission drops depends on the amount and type of filler.

COST/BENEFIT ANALYSIS

A cost/benefit comparison of materials used in conductive polymer composites versus conventional solder is shown in Table 2. Graphite is the conductive material in the composite rather than the more common silver flake filler.

TABLE 2. Comparison of Materials Costs of Graphite-Based Conductive Polymer Adhesive and Tin/Lead Solder

	Material	Cost/lb (\$)	Cost/g (\$)	Density (g/cc)	Cost/cc (\$)
Α	Graphite fibers	17.50	0.039	2.0	0.078
В.	Particulate graphite powder	1.30	0.0029	2.25	0.007
C.	Conductive graphite fiber/powder composite (45% fiber)	10.00	0.022	1.6	0.035
<u>D.</u>	Tin/Lead (63%/37%)	90.00	0.020	8.17	0.16
E.	Savings per cc: (D-C) (D-B)				0.125 0.153

Assuming that a typical circuit board requires approximately 6 cubic centimeters of solder to complete all connections, and that a typical production run would consist of about 1,000 boards, the cost savings by the switch from tin/lead to a graphite powder-filled conductive polymer composite would be about \$150, as indicated in line E. The savings from the switch to the higher conductivity graphite filament would be about \$125, also shown in line E. For a more effective composite, the powder and fibers should be combined for optimum packing. This should provide a materials cost savings for the 1,000 boards of between \$125 and \$150. While this figure may not represent a large savings for 1,000 boards, significant savings may be realized over large production runs or when a larger volume of solder per board is required. Environmental benefits include the elimination of lead and heavy metals from the process; elimination of solvents used in conventional solder methods; and freedom from flux residue on the boards that would require solvents plus labor costs to clean.

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Incentives

Polymer based solder material is a practical substitute for metal-based solders. Additional development work will be necessary to define all parameters for achieving high electrical conductivity and defining appropriate manufacturing and application methods. Tests indicate that other metal replacement opportunities exist for such applications as antennas and electromagnetic shields.

Large or small organizations that are producing or fabricating electronic parts may be able to replace their metal-based solder material with filled polymer systems, depending on the resistance requirements of the applications.

Barriers

The polymer solder material may not be as universally accepted as the traditional tin/lead-based solder, mainly because the higher conductivity of metals (resistance 0.4 to 0.6 ohm) in comparison with the conductive polymer composite coatings and adhesives (resistance 5 to 10 ohm). Also, at this stage of development, the fillers are not dispersed as uniformly as will be desirable for many future applications.

Potential Solutions

Much higher conductivity of these polymer-based solder materials can be achieved by additional selection and optimization of filler ratios, but the present product can probably be used in capacitors and high voltage applications where very high conductivity is not required. Further investigations with improved dispersion methods, such as twin screw extruders and Banbury mixers, should be conducted.

COMPOUND ADIABATIC AIR CONDITIONING FOR TRANSIT BUSES

by

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ABSTRACT

Adiabatic air conditioning (AAC) is a water-based cooling process that requires minimal energy and uses no refrigerants, such as chlorofluorocarbons (CFCs). Compound adiabatic air conditioning (CAAC) is an advanced, two-stage version of the same general process, using indirect-direct cooling technology. In the indirect stage, air is cooled without increasing its moisture content. During the direct stage, the air is cooled further by evaporating moisture into the air stream. The operable chemical is each phase is water.

This project assessed the performance of a CAAC system installed on a bus and operated under a variety of climate conditions representative of summer design conditions in selected metropolitan areas.

INTRODUCTION

PROJECT DESCRIPTION

Air cooling is a physical process governed by the laws of thermodynamics. The relationships between air temperature and water content are well known and are described on psychrometric charts, which provide a foundation for understanding air conditioning performance.

In a traditional *refrigerant air conditioning* system, a refrigerant gas is circulated in a closed system. The gas is pressurized and liquified by a compressor. When the pressure is released, the liquified refrigerant quickly evaporates, absorbing heat from its surroundings (an evaporator coil). An air stream is passed through an evaporator coil and the air is cooled sensibly, meaning that no moisture is added. The absorbed heat is carried by the gas until it can be rejected in a second process (a condenser coil). A typical system contains two independent sets of blowers, motors and coils, associated components, and a compressor.

In an adiabatic, or evaporative, air conditioning (AAC) unit, air and water are mixed, and the water evaporates, absorbing heat from its surroundings. This process occurs naturally, and pressurization is not required; the energy requirements are thus much lower than those of a refrigerant unit. The amount of cooling accomplished depends on the moisture content of the incoming air. Dry air can hold more water, so more cooling can occur. Humid air already contains some moisture and thus has less evaporative potential. Evaporative cooling has been limited to dry climates for this reason.

A compound adiabatic air conditioning (CAAC) system treats the supply (incoming) air in two stages. In the first stage, the supply air is cooled sensibly. In this project, a crossflow, wet-surface/dry-surface heat exchanger was used (although other methods can be employed). On the wet side, air and water are mixed, the water evaporates, the air temperature is reduced, and the humidified air is exhausted to the atmosphere. Supply air is passed through the dry side, its heat is absorbed through the heat exchanger walls, and its moisture content is unchanged.

In this process, the dry bulb and wet bulb temperature of the supply air are both reduced, so that the leaving air has an entirely new set of conditions that increase the overall evaporative potential.

The cooling capacity of any air conditioning system is expressed by the following formula:

BTUh = (CFM) $(1.08 \times \text{elevation factor})$ (delta T)

where:

- BTUh = cooling capacity, or load
- CFM = air volume in cubic feet per minute
- elevation factor = 1.00 at sea level (lower at altitude)
- delta T = difference between the temperature of the air entering and leaving the subject space

For example, on a bus where the entering air is 60°F and the return (exhaust) air is 80°F, the delta T is 20°F. If the air conditioning unit delivers 3,000 CFM, the cooling capacity is 64,800 BTUh.

Transit buses present an extremely challenging cooling situation and require very large, energy-intensive refrigerant air conditioning systems. This is due to high occupancy, large glass surfaces, engine heat, and extreme infiltration from opening and closing doors. Low energy CAAC systems can provide equivalent cooling along with greater air volume and air movement, both desirable features. The technical challenge is to maintain acceptable performance under moderate and high humidity conditions.

The two-stage cooling process used in the CAAC system can supply air to a given space that is both cooler and drier than what is possible with a single stage AAC system. This capability can make it possible to maintain comfort in higher humidity conditions and could substantially increase the geographic range where this low energy, CFC-free technology can be successfully employed.

Unique Process Features/Advantages

The Climatran CAAC system has two main elements: the actual cooling unit that mounts on the rear of the bus, and a water tank. The main cooling unit is located in the upper rear area of the bus where one might expect to see a rear window. This unit consists of two independent sets of blowers and motors, a water circulation pump and distribution system, an electrical control panel, rigid evaporative media, and a crossflow plate heat exchanger. The water tank can be mounted on the bus

underbody near the front axle; the tank supplies water to the cooling unit with a transfer pump that is activated by a liquid-level switch in the cooling unit.

The unique component is the crossflow, wet-surface/dry-surface heat exchanger that pre-cools the supply air without increasing the moisture content. This is called "sensible" cooling. As the dry bulb temperature of the air is reduced, the wet-bulb temperature is also reduced proportionally.

After the air leaves the heat exchanger, it is passed through an adiabatic cooling stage where water and air are mixed and evaporation occurs. The air temperature is reduced in proportion to the amount of water evaporated. If the incoming air is dry, more water can be evaporated, resulting in greater cooling. In an adiabatic system, the air temperature will be reduced until the air reaches saturation, at which point the air temperature will equal the wet-bulb temperature of the entering air.

The heat exchanger serves two functions. As noted above, the crossflow heat exchanger reduces both the ambient wet-bulb temperature and the dry bulb temperature. Hence, the second stage, adiabatic cooler receives air that is cooler than the ambient air and has a lower wet-bulb condition that results in greater cooling potential.

The unique attributes of a CAAC system produces adequately cool air in climates otherwise unsuitable for standard evaporative air conditioning, thereby eliminating the use of CFCs and using little energy.

APPLICATION

Process Replaced

Motor vehicle air conditioning systems have traditionally contained CFC-12 refrigerant, commonly known under the trade names "freon" and "R-12." This chemical has been one of the major causes of stratospheric ozone depletion and contributes to global warming. EPA identifies CFC-12 as a class I ozone-depleting substance. CFC-12 can be operated in equipment using flexible, rubberized tubing.

In recent years some applications, such as transit buses, have begun using HCFC-22 (hydrochlorofluorocarbon), a somewhat different chemical compound, but with properties similar to CFC-12. Although less destructive than CFC-12, HCFC-22 still releases chlorine molecules that interact with and destroy ozone molecules in the stratosphere; it is also a potent "greenhouse" gas. EPA identifies HCFC-22 as a class II ozone depleting substance. This compound is very elusive and difficult to contain, and equipment using HCFC-22 must use rigid copper piping that can be subject to leakage. HCFC-22 has greater cooling capacity than CFC-12 and it, too, is scheduled for a production phase-out under the Montreal Protocol and the Clean Air Act Amendments.

Traditional air conditioning manufacturers have recently introduced a refrigerant designated as HFC-134a (hydrofluorocarbon) that contains no chlorine atoms and may not destroy ozone molecules. HFC-134a has less cooling capacity than either

CFC-12 or HCFC-22 and is more difficult to contain and maintain, which may result in excessive, unintentional discharges and resultant consequences.

Wastes Prevented

Although new regulations require mechanics servicing air conditioners to use recycling equipment, refrigerants still may leak during normal unit operation. Leakage is the single most common reason that equipment is serviced. The unintentional release of ozone-depleting chemicals is likely to continue, despite the use of recycling equipment.

Refrigerant air conditioning is also very energy intensive. A typical bus air conditioning system operates in a range between 15 to 30 horsepower; 21 horsepower is considered as the standard value. A study by the Federal Transit Administration found that refrigerant air conditioning results in an increase in fuel consumption of about one gallon per hour of operation. Depending on the operating area, this can amount from 600 to 4,500 gallons per bus per year.

Since adiabatic systems operate on about 85 percent less power than refrigerant equipment, substantial fuel savings are possible, which would reduce carbon dioxide emissions as well as other combustion by-products and particulates.

Cross Segment Uses

CAAC systems are already available for residential, commercial, and industrial applications. This technology could be applied to trucks, cars, and other transportation vehicles.

PROCEDURE

DEMONSTRATION

After the CAAC system was installed on the test bus, the bus was driven to a variety of locations to conduct performance tests under different climate conditions. The 1992 summer weather was unusually mild in many parts of the U.S., which complicated route selection. The main goal was to conduct some tests at dry bulb temperatures around or above 90°F with coincident wet bulb temperatures of about 72°F or higher. Such tests would be representative of typical design conditions in most of the northcentral, northeastern, and middle Atlantic states.

The original plan to test the bus in cities such as Chicago, Detroit, Pittsburgh, New York, and Philadelphia became impractical due to the mild weather. Instead, on the first trip, the bus was driven from Denver eastward to Salinas, Kansas, then southward through Witchita and Oklahoma City. The return trip went west through Abilene, north to Lamar, and back to Denver, a total of 1,671 miles. The itinerary for the second trip also began in Denver, proceeded south to Albuquerque and El Paso, and west to Tucson and Phoenix where the demonstration was completed after 1,498 miles.

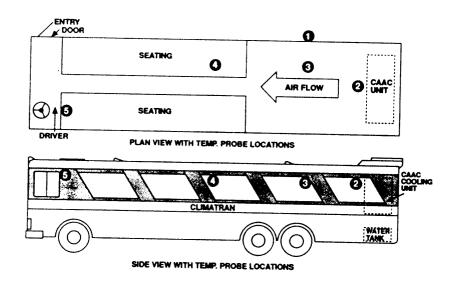
Outside temperature and humidity measurements were taken at irregular intervals and, when suitable, a complete cooling performance test was conducted. A total of thirteen cooling tests were conducted. During each of these tests the bus would remain stationary, reducing the number of variables.

The vehicle was equipped with thermocouples at five fixed points:

- Probe 1 measured the ambient dry bulb temperature
- Probe 2 measured the supply air temperature leaving the CAAC system
- Probe 3 recorded the interior space condition in the rear of the coach
- Probe 4 recorded the condition in the middle of the coach
- Probe 5 recorded the condition at the driver's seat

The probe locations were identical to those in prior tests (separate from this project) of an AAC system. Figure 1 shows the top and side view of the bus with temperature probe locations.

Figure 1. Top and Side View of Test Bus with Temperature Probe Locations



After recording conditions with the air conditioning system off, the tests were begun by activating the CAAC system and recording temperatures at the 5 probe locations at 2 minute intervals while the bus remained stationary. The tests were continued until interior conditions stabilized. Temperatures were recorded on a multichannel Electrotherm 600. Wet bulb temperatures were measured with a Pyschrodyne psychrometer.

EVALUATION PARAMETERS

There is a difference between refrigeration and comfort air conditioning: the former process is intended to maintain low temperatures to prevent spoilage, and the latter is intended to maintain human comfort. Either may be used in process applications or to maintain specialized environments.

Comfort

Vehicular air conditioning is intended to maintain human comfort for passengers. However, the notion of "comfort" is elusive and subject to many variables. Four basic elements affect human comfort: temperature, humidity, air velocity, and air volume. Other factors include clothing, activity level, length of exposure, individual transpiration rate, and individual perceptions.

The American Society of Heating, Refrigeration, and Air Conditioning Engineers (ASHRAE) has developed conditions that generally describe the human comfort zone in terms of temperature and humidity. In his book, *The Evaporative Air Conditioning Handbook*, Dr. John R. Watt published a revised comfort zone that also considers air velocity. University studies have addressed human perceptions of comfort. Unfortunately, in practice, little regard is given to such quantifications of comfort; instead the operable policy appears to be the colder, the better.

According to studies conducted for ASHRAE, most human comfort occurs between 70° and 80°F. Fewer people are comfortable at either extreme of this range and the greatest number of people experience comfort between 74° and 76°F. Additional studies by Kansas State University (KSU) have found that if humidity levels are high, lower temperatures are needed to maintain comfort, and if humidity is low, people can remain comfortable at higher temperatures. If air velocity or air volume is low, lower temperatures are needed to maintain comfort, and if they are high, comfort can be maintained at higher temperatures. The KSU studies also found that human tolerances to extreme conditions are greater when exposure is for shorter time periods, and that perceived discomfort increases over time.

In this project, the cooling performance of the CAAC system was measured against both the ASHRAE and the Watt comfort zones. The ASHRAE Comfort Zone indicates that human comfort occurs between 70° and 80°F with 20 to 60 percent humidity. The Evaporative Cooling Comfort Zone, described by Dr. John R. Watt, also considers the cooling effect of air velocity and generally indicates that human comfort occurs between 70° and 83°F with 20 to 80 percent relative humidity with appropriate air velocities. The humidity aspect presents some distinct problems. In any type of air conditioning system, the humidity will vary significantly throughout the space, making it almost impossible to address quantitatively. Therefore, it will be addressed qualitatively, which, admittedly, does little to convince skeptics.

Design Conditions

Since climate conditions vary, equipment or application engineers must consider these conditions when designing and sizing an air conditioning system. ASHRAE developed and maintains data describing the historical design conditions for many areas.

Design conditions are expressed in terms of dry bulb and wet bulb temperatures. They are presented to give the design engineer flexibility in selecting the appropriate configuration. In most cases, a system that can maintain comfort 100 percent of the time is considered inefficient and overly expensive, since extreme conditions are relatively rare. ASHRAE design conditions are expressed in terms of the percentage of ALL hours that occur at or below the given design condition.

In a laboratory, the maintenance of specific atmospheric conditions might be imperative. In an office, productivity might require that comfort be maintained almost all of the time. On transit buses or other passenger vehicles, comfort is desirable, but not crucial to operations or productivity. Therefore, 2.5 percent dry bulb and 5 percent wet bulb design conditions will be considered as standards.

ASSESSMENT SUMMARY

This project has shown that a CAAC system can maintain a comfortable environment in moderately high humidity conditions. However, insufficient data were accumulated to facilitate comprehensive documentation of cooling performance under a wide variety of conditions. Since the test vehicle lacked adequate electrical power to operate the CAAC system at its designed output, performance results are not conclusive; in-service performance would have to be predicted from extrapolation of the results. Although this is certainly possible, this approach could be unlikely to convince skeptics that a CAAC system would be feasible in their particular location.

COST/PAYBACK OF DEMONSTRATION

The cost of the project was \$57,077.13, with \$25,000 provided by EPA through the Pollution Prevention By and For Small Business Grant Program, and the balance of \$32,077.13 contributed by Climatran.

The following estimates of fuel and cost savings are based on a report published by the Urban Mass Transit Administration (UMTA). Assuming that a U.S. Government bus operates for about 1,500 hours during the cooling season, fuel savings could approximate 1,500 gallons per year, or about 1 gallon per hour of operation. UMTA reports that typical annual maintenance costs for city bus air conditioning range from \$1,200 to \$1,700, in 1982 dollars. Considering inflation and the added costs related to compliance with the Clean Air Act Amendments of 1990, maintenance cost savings could exceed \$2,000 per bus, per year. The combined savings would be about \$3,500 per bus, per year.

RESULTS AND DISCUSSION

PERFORMANCE RESULTS

During the project, thirteen individual cooling performance tests were conducted. Ten tests assessed the CAAC system, while three tests were conducted only on the AAC component. All tests were performed with the bus stationary and the engine running. Prior to activating the CAAC system, temperatures were recorded for each of 5 probes, including ambient dry-bulb and wet-bulb conditions.

The tests were begun by activating the CAAC system and recording temperatures for each probe at approximately two-minute intervals. The tests were continued until interior conditions stabilized. Each test was identified by a number and location.

The performance assessment was based on the average interior temperature achieved at the end of each test period and on the actual temperature achieved at

each probe location. The average interior temperature is the average temperature recorded for probes number 3, 4, and 5. In evaluating temperatures at specific locations, the key is probe number 5, which is at the driver's seat and furthest from the incoming supply air.

In all but one test, the CAAC system maintained an average interior temperature within the traditional ASHRAE comfort zone. In test Number 9, the average interior temperature (81.7°F) exceeded the upper limit of the ASHRAE comfort zone (80.0°F), however the average temperature did fall within the comfort parameters of the Watt comfort zone.

Test Number 9 was conducted at Billings, Oklahoma with an ambient dry bulb temperature of 90°F and a coincident wet bulb temperature of 75°F. Measured air flow was 2,400 cubic feet per minute. The supply air temperature was 75.4°F, and the maximum exhaust air temperature was 83.5°F. Calculated cooling capacity was 21,000 BTUh. If air volume was increased to the design point of 4,000 cubic feet per minute (by installing a larger alternator), the cooling capacity would increase to about 35,000 BTUh.

Since a 21,000 BTUh unit reduced the average temperature by 8.56°F, it could be calculated that a 35,000 BTUh unit would reduce the average temperature by 14.3°F. The exhaust temperature as measured on probe number 5 would be reduced from 83.5°F to about 76.6°F. The CAAC system, if operating as designed, could easily have maintained comfort under either comfort criteria.

In referring to the formula for cooling capacity, one may see that the only way to maintain the condition of a given space as the supply air temperature rises is to increase the air volume. Given sufficient air volume, comfort temperatures can be maintained at virtually any ambient dry bulb design condition in the U.S.

Based on Climatran's analysis of the results of this project, a CAAC system can maintain comfort at design conditions up to, and including 90°F dry bulb temperature with a coincident wet bulb temperature of 75°F. Increasing the air volume of the CAAC system of 4,000 cubic feet per minute would increase cooling capacity providing further assurance that comfort would be maintained at 75°F wet bulb temperature and extend the dry bulb parameter significantly above 90°F. The CAAC system could maintain interior comfort conditions at 95°F dry bulb temperature, 75°F wet bulb temperature, and possibly somewhat higher.

The continental United States may be divided into four general regions where AAC or CAAC technology could be successfully applied to transit operations, based on the performance measured during this project. Zone I, which includes most of the western U.S., is characterized by wet bulb temperatures less than 70°F. Zone II includes a narrow area in the midwest and most of the northcentral and northeastern states. This region has wet bulb temperatures of 70° to 73°F and moderate humidity. CAAC systems could be appropriate, but lower-cost AAC equipment also could be used in applications where occasional reduced cooling performance is tolerable. Zone III, a high-humidity area in the southcentral U.S. that includes Virginia, Kentucky, North Carolina, Tennessee, Missouri, and parts of Texas, is defined as areas having wet bulb temperatures of 74° to 75°F. A CAAC system would be feasible; AAC systems are unlikely to be acceptable, except for short periods. Zone IV covers the

deep southeastern states where very high humidity results in wet bulb temperatures of 75°F and higher. The cooling performance results in this project were not evaluated under conditions representative of Zone IV weather, and thus did not prove that a CAAC system could maintain comfortable conditions at 75°F wet bulb temperature and higher. However, comfort might be maintained at or above 75°F wet bulb temperature if the system air volume was higher than that tested in this project.

PRODUCT QUALITY VARIANCE

Product quality, considered as cooling capacity and efficiency, was maintained throughout the test program, although not at the performance level to which the equipment had been designed. The equipment suffered no mechanical failures, and cooling performance was consistent relative to expectations under the variety of ambient conditions encountered. The supply air volume was significantly less than designed. Although the supplied air temperatures were acceptable, the volume of air delivered to the vehicle interior was much lower than planned.

Since cooling capacity is a function of both air temperature and air volume, the CAAC cooling capacity was significantly lower than designed. Therefore, interior temperatures during testing were higher than they would have been if the air volume had been as designed.

After each stationary cooling test, the bus was put in motion. The temperature at the driver's seat became more comfortable, and air movement became more noticeable. After installing a supplemental thermometer, the driver was able to monitor the temperature at the driver's seat while in motion, which confirmed that the temperature decreased further when the bus was in motion.

In test number 11 (Tucson, Arizona), the driver's area reached a temperature of 79.9°F during the stationary test. When the bus began to move, the temperature at the driver's seat averaged 73.5°F.

CONDITIONS THAT IMPACT PERFORMANCE

The CAAC air volume was measured and calculated at 2,400 cubic feet per minute. However, the equipment was designed to supply 4,000 cubic feet per minute. System components were selected to perform at a level that would supply the desired air volume.

The power supply from the existing bus alternator was 145 amps, while the power demand from the blower motors was 255 amps. The actual power available was only about 60 percent of that necessary to operate the system as designed.

The air volume was measured at 2,400 cubic feet per minute compared to the designed output of 4,000 cubic feet per minute. This correlated directly with the shortage of electrical power.

After this situation was identified, a new, higher capacity alternator was ordered. Unfortunately, the alternator did not arrive before the weather had deteriorated to cooler temperatures. The alternator was not replaced, as no further testing could be conducted.

While the bus was in motion, the location of the test equipment prevented the driver (the only person on the bus) from monitoring the various temperature probes, with the exception of the supplemental thermometer measuring probe number 5.

The CAAC unit included a roof top deflector, or hood, to keep rain from entering the unit. This hood had an air inlet opening that faced the front of the bus. This enhanced the performance of the blowers and offset the shortage of air volume (that resulted from the under-sized alternator).

Future testing should document the effects of the air inlet and evaluate cooling performance of the CAAC system when operating at its designed output. Such increases in air volume would substantially enhance cooling performance and could result in the ability to maintain comfort at and above 75°F wet bulb temperature.

TABULATION OF DATA

The results of the 13 individual cooling tests are summarized in Table 1 and shows the outdoor conditions during the tests and the cooling results in terms of the average interior temperature. To give perspective to the ambient climate conditions, the last column indicates cities having summer design temperatures similar to the conditions experienced during the test.

TABLE 1. Summary of Cooling Performance Test Results

Test Number	Test Type	Ambient dbt/wbt*	Ave. Interior Temp. (°F)	Location
1	AAC	88/61	77.30	Denver
2	CAAC	88/61	69.90	Reno
3	CAAC	91/67	75.30	Pasadena
4	CAAC	88/74	79.60	New York
5	AAC	89/67	80.10	Burbank
6	CAAC	90/67	76.10	Amarillo
7	CAAC	84/69	75.90	Buffalo
8	CAAC	85/66	75.30	Portland, OR
9	CAAC	90/75	81.70	Atlanta
10	CAAC	90/73	79.10	Philadelphia
11	AAC	93/62	77.00	Salt Lake
12	CAAC	96/61	75.00	Albuquerque
13	CAAC	104/62	73.40	Tucson

^{*}dbt/wbt - dry bulb temperature/wet bulb temperature

COST/BENEFIT ANALYSIS

In the report, "Evaporative Coolers for Transit Buses," published by the Federal Transit Administration (formerly UMTA), the University of Denver, Denver Research Institute conducted a life cycle cost comparison between refrigerant and evaporative air conditioning equipment.

This report concluded that substantial fuel savings and maintenance cost savings could be realized with evaporative equipment. Fuel savings would amount to approximately 1 gallon per hour of system operation. Maintenance costs would be about \$200 per year compared to \$1,200 to \$1,700 per year for a refrigerant-equipped bus. Although the reliability of an evaporative unit was found to be substantially greater than a refrigerant unit, no additional cost savings were attributed to this factor.

A vehicle operator could expect maintenance cost savings of about \$1,250 per bus per year with an evaporative system. Since this report was published, EPA has required CFC recycling and prohibits known releases of CFCs. This would likely increase maintenance costs for CFC-based equipment above the levels noted in the FTA report.

Operating hours range from 600 to 4,500 hours per year depending on geographic regions and the type of service provided. Assuming 1,800 annual operating hours over a 5 month period, an operator could save 1,800 gallons of fuel (at \$1.00 per gallon) per bus, and about \$1,500 on maintenance, for a combined annual savings of \$2,300 per bus equipped with an adiabatic system.

On new buses, the purchase price of CFC systems and AAC systems are virtually equal, however adiabatic equipment could generate immediate savings due to lower fuel and maintenance costs. The purchase price of CAAC units would be approximately \$1,500 more than either the CFC or AAC units; given monthly savings of about \$500, the simple payback period for a CAAC system would be about 3 operating months.

Metropolitan transit operators receive federal subsidies for equipment purchases and operating shortfalls. These subsidies are traditionally 80 percent of cost. The operator's share of the CAAC premium is only 20 percent, or about \$300, which would payback in less than one month. Both the transit operator and the federal government could save money by adopting the AAC and CAAC systems.

Existing buses generally operate CFC-12 air conditioning equipment and experience substantial leakage and major maintenance expenses, much of which is equipment replacement. Retrofitting existing vehicles could immediately reduce CFC emissions. A bus could be converted to a non-CFC system for about \$9,000 (subsidized by \$7,200 in federal funds). Annual operating cost savings would be about \$2,300 or more, since CFC equipment maintenance costs are higher as equipment ages, and retrofits would be done to older vehicles.

The local operator could recover its \$1,800 investment in about 4 months, while the entire retrofit cost could be recovered in about 20 operating months.

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Incentives

This project showed the potential of CAAC systems to provide comfort air conditioning for transit buses under climate conditions that would have been impractical for traditional evaporative cooling equipment.

The CAAC equipment produced for and tested under this program functioned with no mechanical or electrical failures, but did not perform as designed due to shortage of electrical power on the test vehicle. Despite this significant deficiency, the CAAC system was able to maintain interior comfort conditions as defined by one or both sets of criteria designated for use in this evaluation.

Cooling performance data were collected during 13 individual cooling tests conducted under a variety of climate conditions. Testing showed that the cooling capacity of the CAAC system is inversely related to outside humidity (wet bulb temperatures). Based on the data collected, the CAAC system has the capacity to maintain comfort conditions at ambient conditions of at least 90°F/73 percent humidity. Since cooling performance suffers little from increasing dry bulb temperatures, comfort can likely be maintained up to 95°F/73 percent humidity.

Although cooling performance was affected by a shortage of electrical power from the bus alternator, one may calculate cooling capacity that could have resulted using a properly sized alternator and extrapolate expected performance based on actual test results. In this case, the CAAC system could easily have maintained comfort at the above-mentioned conditions.

Based on the demonstrated cooling capacity of the CAAC system, this technology could be employed in areas where design conditions are appropriate, which would include all of the continental U.S., except for the general region east of 100 degrees west longitude and south of the 36th parallel, with other localized exceptions.

Adoption of this technology could eliminate the use of ozone-depleting CFCs from bus applications and would conserve fuel.

Barriers

Barriers to the advancement and acceptance of this technology are primarily political in nature, rather than technical. Technical improvements and cost reductions could be expected to coincide with market development, however AAC and CAAC systems already offer substantially lower life cycle costs than CFC-based refrigerant systems. Opportunities exist for the federal government to adopt this technology and to encourage its use by federally subsidized metropolitan transit operators.

The cost of a CAAC system is currently about 25 percent higher than that of a CFC system. CAAC technology has no scale economics.

Transit operators receive subsidies from the Federal Transit Administration (FTA) for equipment purchases and operation shortfalls. This policy removes any economic incentive to purchase cost-effective equipment or to operate more efficiently. CFC equipment becomes more affordable and is often used in areas where the need for air conditioning is marginal.

FTA refuses any assistance to AAC technology and has imposed testing requirements that block the use of this technology on new buses. While the Department of Defense has approved the use of AAC technology, the General Services Administration will not specify the inclusion of such equipment on new vehicles, citing an executive order by President Reagan that minimized unnecessary requirements on private businesses supplying the federal government.

The crossflow heat exchanger currently in use is the only model available. The efficiency of this device could be higher, but no manufacturer produces an improved version, since there is virtually no market for it. A higher-efficiency heat exchanger would result in lower supply air temperatures and lower humidity levels, both of which would enhance performance and possibly reduce equipment size and cost.

The configuration of the heat exchanger is such that it cannot be used in a roof-mounted unit, as it is fairly tall.

The packaging of the components within the cabinet is complicated and results in added sheet metal expense.

Both AAC and CAAC systems cool by increasing the humidity above ambient levels. In very high humidity conditions, this adversely impacts interior comfort and requires very high air volume levels to maintain acceptable temperatures.

Potential Solutions

Although the purchase price for CAAC equipment is currently about 25 percent higher than for CFC equipment, the technology has no market share, no economies of scale, and no competitive pressure. As the market for this type of equipment is developed, the first cost premium could be eliminated quickly through natural economic factors.

The present subsidy program may discourage transit operators from pursing cost-saving measures and encourages the use of CFC equipment. Through subsidized costs, CFC systems become more affordable and are often used in areas where the need for air conditioning is marginal.

One approach to this dilemma is to amend the subsidy formula to remove any subsidy for the purchase of CFC and HCFC equipment, while retaining funding for safer technologies. An alternative plan would require grantees to factor a life cycle cost adjustment into their bid price comparisons. In this approach, bid prices would be evaluated on the overall life cycle cost, not just the purchase price; hence equipment/vehicles with long-term cost savings potential would have a competitive edge. FTA could also require the use of a cost adjustment factor for environmentally preferred equipment. This could apply to components other than air conditioning systems, such as alternative-fuel engines.

REDUCING HEAVY METAL CONTENT IN OFFSET PRINTING INKS

by:

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ABSTRACT

Ecoprint created a commercial offset printing ink using pigments with no heavy metals, thus preventing pollution in three key areas: (1) in the waste ink produced by a printer that must be handled as a hazardous waste; (2) in the printed materials that are landfilled or incinerated; and (3) in the sludge that is created during the de-inking and repulping of waste paper fibers as they are made into recycled paper. The result of the testing throughout the project was the creation of a "palette" of colored inks with a low heavy metal content.

INTRODUCTION

PROJECT DESCRIPTION

Printing is an \$80 billion a year industry in the United States and uses large amounts of ink. Colored inks contain many heavy metal-based pigments that are applied to a myriad of printed materials that ultimately end up in the nation's waste stream. This project had five key components:

- Tested the 11 primary colors of printing inks to determine the concentration of 12 key metals
- Identified alternative pigments that were not based on heavy metal compounds
- Formulated new offset printing inks based on these non-heavy metal pigments
- Tested these inks in actual commercial printing conditions
- Created a "palette" of non-heavy metal-based inks in primary mixing colors

Unique Product Features/Advantages

These non-heavy metal-based offset printing inks can be successfully used in most commercial printing applications including the printing of brochures, newsletters, direct mail promotions, letterhead, and many other products. Ecoprint knows of no other sheet-fed lithographic inks on the market that have tested as low for metals content.

APPLICATION

Products Replaced

The result of this project is the creation of inks with greatly lowered heavy metal content, thus preventing pollution in three key areas: (1) in the waste ink produced by a printing company that must be handled as a hazardous waste; (2) in the printed materials that are later landfilled or incinerated; and (3) in the sludge that is created during the de-inking and re-pulping of waste paper fibers on their way to being made into recycled paper.

Waste Prevented

While Ecoprint cannot foresee direct cost savings to the printer (waste ink of all kinds still contains oily compounds, and as such will have to be disposed of as hazardous waste), but there will be savings on a more general, societal level if the use of these new inks becomes commonplace. These savings will come in the form of significantly reduced pollution of air, water, and land. Incineration of waste papers printed with these new inks will produce ash and air emissions that are lower in copper and barium.

Cross Segment Uses

Other sectors of the printing industry also may be able to use these inks, although they would need to be reformulated to be compatible with the different machinery used. Additional applications include flexographic printing, engraving, silkscreening, and other types of offset printing such as web printing (cold and heatset). A wide variety of products -- from packaging materials to magazines -- are printed with these different printing applications.

PROCEDURE

DEMONSTRATION

Ecoprint first selected the metals to be tested by surveying a number of expert sources -- EPA's 33/50 Program, The Chesapeake Bay Foundation, and the National Toxics Campaign Laboratory -- for information on environmental toxins. The 12 target metals selected were:

- Antimony
- Arsenic
- Barium
- Cadmium
- Chromium
- Copper
- Lead
- Mercury
- Nickel
- Selenium
- Silver
- Zinc

Ecoprint then arranged for the testing of the 11 primary ink colors (neutral black, transparent white, reflex blue, process blue, green, yellow, warm red, rhodamine red, rubine red, purple, and violet) to determine which inks had problematic heavy metal contents. Samples of the 11 inks were sent to the National Toxics Campaign Laboratory in Boston where the inks were tested for heavy metal content with a Perkin-Elmer Atomic Absorption 5100 spectrophotometer. Certain samples were further tested for barium using Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), when false readings were generated by the presence of calcium.

Ecoprint selected the target ceiling of 100 parts per million (ppm) for a heavy metal content goal. Tests revealed the problem ink colors to be reflex blue (over 200 ppm copper), process blue (3,800 ppm copper), yellow (859 ppm zinc), green (3,300 ppm copper), warm red (122 ppm barium), rubine red (150 ppm zinc), and rhodamine red (181 ppm copper). The other colors tested below the 100 ppm level for the target metals.

Ecoprint then experimented with alternative, non-heavy metal-based pigments, such as pigments based on calcium compounds or organic pigments. These alternative pigments should match existing colors as closely as possible and should be compatible when mixed with the other components of the ink, such as the resins and oils.

Since these reformulated inks are a proprietary product that will be subsequently marketed, the alternative pigments used cannot be specifically named. However, the pigments are sold by well-known pigment manufacturers, but have not generally been used in printing ink formulations.

The actual mixing of the inks was conducted by Ecoprint's subcontractor, Alden and Ott Inks. Newly formulated inks that contained the alternative pigments were then sampled and sent back to the laboratory to again test for metal content, again with the goal of formulating inks that tested below 100 ppm for each of the 12 metals. Several colors of inks were reformulated in one trial; other ink colors required several trials to obtain the correct formulation. Only two colors, rubine red and rhodamine red, could not be reformulated, as pigments could not be found to match the shade of these colors.

Once achieving the desired test results for metal content, the inks were tested on a printing press to determine their printability. As the inks were used in printing, their performance was monitored in several areas: drying time; absorption onto the paper surface; compatibility with printing plates, fountain solutions, cleaning agents, and solvents; and "holdout" on the sheet after printing to determine if density of color remained strong. Inks were also mixed to see if a sufficient number of different colors could be created to satisfy most commercial requirements.

Approximately 12 press tests were conducted on a Komori Sprint, 1978 model, 2-color press. The machine was retrofitted with an Epic Delta Dampening System, which utilized a separate water form roller with an oscillating bridge roller that contacts the first ink form roller. Other specifics included:

- Running speeds: Between 5,000 to 9,500 sheets per hour
- Blanket: Reeves Vulcan 714 Compressible
- Plates: 3M Viking subtractive
- Fountain Solution: Rosos G-C #1J One-Step concentrate, mixed as follows 6 ounces concentrate, 2 ounces gum arabic, using water from a reverse-osmosis unit; solution recirculated through Royce refrigeration unit
- Room Temperature: Between 68 and 75 degrees F
- Print Run Length: 1,000 impressions up to 20,000 impressions
- Papers: All uncoated and recycled, but varying in weight from 60 pounds offset to 80 pounds cover
- Ink Coverage: From 5 percent to 50 percent coverage, including solids, screens, and traps, as well as line art

The press was set up as for the "regular" inks, with no change in work habits.

Cost of Demonstration

EPA's contribution to the project through the Pollution Prevention By and For Small Business Grant Program was \$25,000; Ecoprint's contribution was \$6,749, for a total project cost of \$31,749.

While the cost of laboratory analysis was higher than first estimated, it was a vital part of the project. Printing compatibility was much more trouble-free than expected, thus lowering costs in this category. Ecoprint was pleased with the overall results of the project, considering its modest scope and the warning by several industry sources that the goals may be "impossible to achieve."

RESULTS AND DISCUSSION

PERFORMANCE RESULTS

Press Test Results

No difference was detected between the reformulated and the original formulation inks in the following categories:

- Interaction with plate surface: No problem was encountered with plate sensitivity in non-image areas, plate blinding, or premature plate wear.
- Mixability of inks: More study would be desirable to determine if pigment strength poses any problem, particularly with colors mixed with a high proportion of transparent white.

- Performance on press: No stripping on ink rollers was seen; wash-up procedures and interaction with fountain solutions (running alcohol-free posed no problems) were normal; and no tendency of inks to "emulsify" was detected.
- Printing on paper: Good absorption onto sheet surface; good trapping with other colors; drying time averaging the same as original formulation inks. Colors seem to be lightfast indoors (i.e., office lighting) for at least two months (and probably much longer, although long term tests were beyond the scope of this project). No tests were conducted where ultraviolet light (i.e., sunlight) was exposed to the printed material for extended periods of time.
- Folding of printed sheets: While occasional marking (where pullout rollers collect ink and redeposit it onto the sheet as a light mark) was noted, this problem was no worse than with the original formulation inks.

TABULATION OF DATA

The metal contents of the five ink colors that were reformulated are shown in Table 1.

Metal Content in Inks before and after Reformulation (Parts per Million) TABLE 1.

	Reflex	Blue	Gre	en	Warm	Red	Yel	low	Proces	s Blue
Metal	Before	After								
Antimony	* <10	<2.5	<10.0	42.00	7.00	<5.0	* <10	<2.0	<10	19.0
Arsenic	6.30	<0.1	<10.0	2.00	17.00	<3.6	4.20	12.00	<0.3	11.0
Barium	8.80	55.6†	76.00	<36.0	122.00	4.50	11.40	<22.0	6.60	29.0
Cadmium	0.35	1,14	0.86	0.50	0.52	3,04	<0.04	<0.1	0.33	0.20
Chromium	<0,2	1,00	8.60	15.90	3.80	12.40	<0.4	<0.07	3,30	11.9
Copper	205.0	1.60	3300.0	10.60	1.22	2.50	2.39	<0.07	3800	9.40
Lead	1.50	1.00	<0.1	<2.0	<0.3	8.90	<0.5	3.60	0.70	<1.0
Mercury	<0.1	<.05	5.10	22.00	0.09	<0.2	1.10	0.73	<0.1	20.0
Nickel	5.28	1.40	4.66	6.60	5.51	1.96	6.08	<0.1	6.53	5.30
Selenium	<10	<0.1	<10.0	<36.0	<10.0	<9.0	<10.0	<22.0	<10	<11
Silver	<0.2	<.05	<0.4	1.20	<0.2	<3.6	<0.2	0.40	<0.2	0.30
Zinc	4.30	10.1	59.00	3.70	26.00	63.20	859.00	9.00	6.20	12.2

^{* &}lt;X indicates that concentration is lower than the limits of detection
† a second test indicated less than 6 ppm

In reflex blue, copper was nearly eliminated, dropping from 205 ppm to less than 2 ppm. Barium did increase to 55 ppm in the first analytical test, but stayed at less than 6 ppm in a second test.

In reformulating yellow, the first analytical test resulted in a reading of 859 ppm for zinc. While the pigment was not changed, Ecoprint suspected that yellow was

being mixed in the presence of another contaminant. A more careful mixing of the yellow did result in the near elimination of the zinc to 9 ppm.

The metals content of the inks that did not require reformulation (as all metals were below 100 ppm) is shown in Table 2.

TABLE 2. Metals Content of Inks not Requiring Reformulation

Metal	Trans. White	Neutral Black	Purple	Violet
Antimony	15.00	<10	<10	<10
Arsenic	17.00	15.00	<7	4.10
Barium	* < 0.5	<0.5	6.60	12.00
Cadmium	0.99	0.67	0.20	0.43
Chromium	2.10	1.10	46.00	2.30
Copper	1.09	4.59	1.40	79.90
Lead	2.10	<0.6	1.30	3.50
Mercury	<0.1	0.50	2.60	<0.1
Nickel	3.80	4.98	4.00	5.16
Selenium	<10	<10	<10	12.00
Silver	<0.3	<0.3	<0.3	<0.2
Zinc	3.10	8.30	8.70	5.10

^{* &}lt;X indicates that concentration is lower than the limits of detection

The metals content of the two inks that could not be reformulated (as alternative pigments could not be found) is shown in Table 3.

TABLE 3. Metals Content of Inks that Could not be Reformulated

Metal	Rubine Red	Rhodamine Red
Antimony	* <10	6.00
Arsenic	0.60	4.10
Barium	90.00	4.30
Cadmium	0.81	0.50
Chromium	4.50	43.00

Copper	2.16	181.00
Lead	3.00	1.30
Mercury	0.01	<0.04
Nickel	6.82	5.78
Selenium	<10	<10
Silver	<0.2	0.07
Zinc	150.00	9.10

^{* &}lt;X indicates that concentration is lower than the limits of detection

COST/BENEFIT ANALYSIS

The reformulated inks are not likely to save printers or their customers money in the printing costs. The inks are likely to be from 20 to 200 percent more expensive per pound in the near term (being produced in small batches). Likewise, there is no cost savings to the printer in waste disposal.

However, while these inks are more expensive, the ink price in relation to the total cost of the printing job must be considered. Inks contribute from 1 to at most 10 percent of a print job's selling price, with most jobs averaging approximately 2 percent. Even a tripling of ink costs would only increase the cost of the average job by approximately 4 percent. In the short term, costs would be slightly higher, particularly because the printer will not be using these inks on all jobs and will order them in small quantities. Like any new product, lower costs can be expected once the product is produced and used in larger quantities.

The cost per pound of traditional inks is compared to the reformulated inks in Table 4.

TABLE 4. Estimated Differnece in Cost per Pound between "Regular" Imks and Non-Heavy Metal-Based Inks (Based on the Purchase of 10 Pound Quantities)

Color	Regular Inks (\$ per pound)	New Inks (\$ per pound)
Reflex Blue	5.85	18.55
Process Blue	5.10	16.15
Warm Red	5.25	11.60
Green	7.15	14.75
Yellow	7.90	9.35

The cost breakdown of a sample print job is outlined in Table 5: an 8 page newsletter, 70 pound opaque recycled paper, 50,000 copies, prints black and one color ink, folded to mail. (These figures are taken from cost breakdowns provided by the Printing Industries of America 1988 "Financial Ratio Study.")

Considering all types of commercial print jobs, ink constitutes, on average, 1.62 percent of the total price of the job. A three-fold increase in ink price would increase the cost of the job by slightly less than 3.4 percent. Small jobs would probably increase by a higher percentage as would jobs using a large amount of ink coverage.

TABLE 5. Cost Breakdown of Sample Print Job

Cost Factor	Price	Percent of Total Cost
Total Sales Price	\$5,000	100.00%
Total materials, including paper, outsider services, but excluding ink	\$1,782	35.64%
Factory payroll	\$1,283	25.66%
Factory expenses	\$664	13.27%
Administration, interest, and selling expenses	\$1,039	20.78%
Profit before taxes	\$151	3.03%
INK	\$81	1.62%

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Incentives

The reformulated inks are now available for use in sheetfed offset printing, and can easily be mixed in larger quantities by Alden & Ott. Since there are many variables in printing and before making wide guarantees of press performance to customers, Ecoprint is conducting even more press tests.

Any sheetfed commercial printing establishment should be able to use these inks. Even web printers (cold-set) should be able to test these inks with some minor modifications in their formulation.

Barriers

Not every color in the Pantone Matching System (PMS) color book can be mixed, as Ecoprint does not yet have substitutes for two primary mixing colors, rubine red (high in barium and zinc) and rhodamine red (high in copper). PMS is the most widely accepted color matching system in the industry, and some customers will need colors that cannot be mixed using non-heavy metal inks.

One solution to the PMS matching dilemma is to continue the research to find substitutes for rhodamine red and rubine red. Should this not be possible, customers can choose from the PMS colors that can be mixed with the 9 colors that all passed the 100 ppm metals test; this still provides a significant palette of colors with which to work.

The cost of some alternate pigments may always be slightly higher, but if these inks were to be adopted by the industry, the price of the pigment may drop due to large-scale production. Print buyers may need to be educated to use less ink coverage on the sheet to control ink costs. For example, one simple way to accomplish this is to discourage designs that cover an area with 100 percent ink and then "reverse out" a headline so that the letters appear in white. Lighter ink coverage has the additional advantage of being "more environmentally sound," as less ink on a sheet makes it more easily recycled.

REUSING ZINC PLATING CHEMICALS

by

Douglas Brothers Global Plating, Inc. Fremont, CA 94538

ABSTRACT

Global Plating, Inc., a full service plating and anodizing job shop, proposed to recover zinc chloride and other chemicals used in zinc plating through ultrafiltration and reverse osmosis membranes. This would reduce the amount of hazardous waste generated, remove chlorides from the wastewater, and permit reuse of the reclaimed metals. Global Plating assessed the effectiveness of the membranes, but technical difficulties prohibited the actual recycling of zinc and plating bath chemicals.

INTRODUCTION

PROJECT DESCRIPTION

Zinc plating of steel is an inexpensive method of protecting steel from corrosion. Zinc plating, when combined with organic brighteners, provides an attractive, bright finish to the workpiece.

In Global Plating's production line, zinc plating solution is dragged out of the zinc tanks on freshly plated parts. The parts are then rinsed with water in the rinse tanks that are overflowed at a rate of 0.5 gallons per minute to maintain a low zinc concentration flow rate and reduce water volume. The overflowed rinse water is piped to a collection sump where it is pumped to a waste treatment system.

In the treatment system, zinc and metals from other plating processes are precipitated from the wastewater. The precipitate is pumped to a filter press where it is formed into cakes 1 inch by 24 inches by 24 inches and containing 30 to 40 percent solids. The cakes are placed in a sludge dryer where water is removed, leaving a powder containing 80 percent solids. This powder is sent to a certified recycler for metals recovery. The discharged wastewater must meet local discharge requirements of 2.6 parts per million zinc.

Global Plating proposed to reclaim the zinc and organic brighteners on-site through the use of reverse osmosis (RO) membranes. This process would allow the materials from the zinc rinse tanks to be returned to Global Plating's process. In plating approximately 5,000 square feet of steel per day, Global Plating uses about 9,000 gallons of water daily for rinsing the plated parts. Zinc levels in the wastewater averages 100 parts per million (approximately 0.75 pounds) of zinc per day.

In the RO system, water is pumped through membranes that allow only the passage of water molecules. Other molecules are rejected and may be recovered. In the zinc plating process, materials that will not pass through the RO membranes are:

- Zinc chloride
- Potassium chloride
- Boric acid
- Zinc brightener
- Zinc brightener carrier
- Organic contaminants
- Inorganic contaminants

The RO membranes can be plugged from contaminants or incompatible chemicals. The correct membrane must be used and contamination must be controlled. Even under ideal conditions, the membranes can become plugged and must be cleaned periodically. Prefiltering the solution to be treated can prevent premature plugging of the membranes. Global Plating installed a 5 micron filter cartridge and ultrafiltration (UF) membranes in the process stream before the solution entered the RO membranes. UF membranes are similar to RO membranes in that they have controlled size pores that allow certain sized particles to pass through. The use of UF membranes extends the life and period between cleanings of the RO membranes. With the addition of the extra filtration, the ratio of zinc concentrate to elutant remained at acceptable levels of above 8:1 for two months after installation of the additional membranes. After two months, the ratio rapidly dropped to 4:1, indicating that the membranes must be cleaned or changed.

Zinc rinse water was passed through the RO membranes that only allow water to pass through. The filtered water was returned to the zinc rinse tanks at 2 gallons per minute. The concentrate was recycled at 16 to 20 gallons per minute in the RO process until the conductivity of the concentrate reached a preset level. At this time, a solenoid valve opened and the concentrate was returned to the zinc plating tank at 2 gallons per minute.

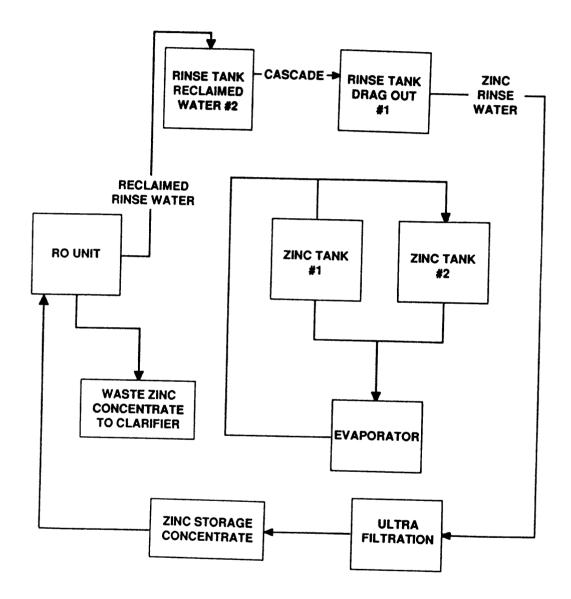
Unique Product Features/Advantages

The RO membranes can recover zinc and the organic brighteners more efficiently than precipitation. The process also can be conducted on-site so that the zinc and brighteners may be returned to the plating process.

Process Schematic

Global Plating's concentrated zinc recovery system is shown in Figure 1.

Figure 1. Concentrated Zinc Recovery System

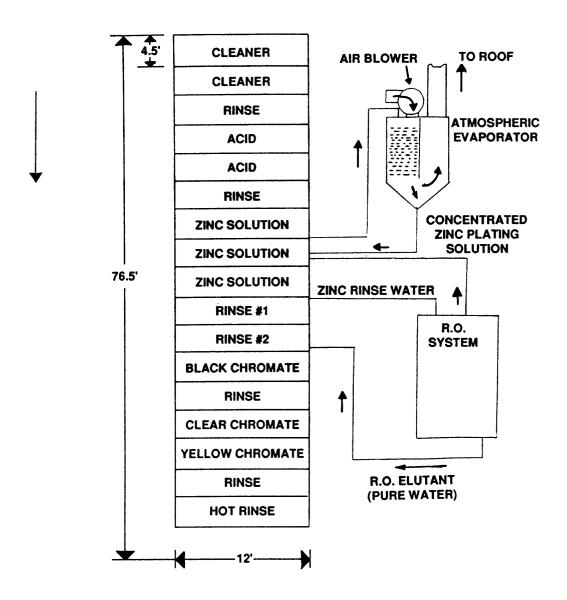


APPLICATION

Process Replaced

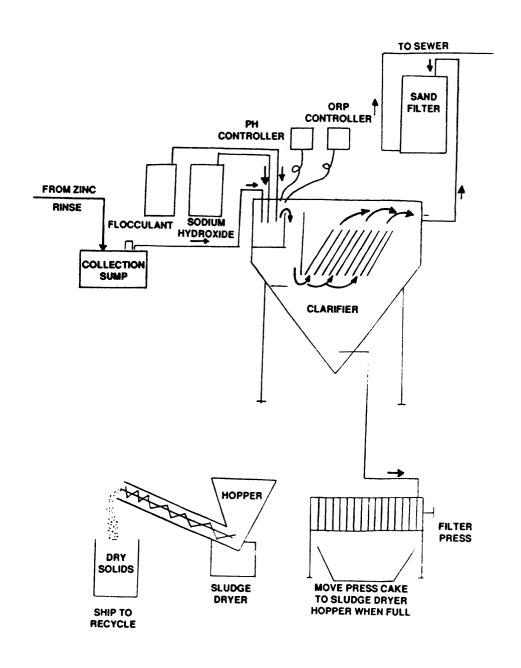
The RO process may be used to replace or reduce the size of a conventional precipitating, clarifying waste treatment system. The conventional clarifying, precipitation system is shown in Figure 2. In Figures 1 and 2, the illustrated process takes concentrated zinc metal from the drag-out tank and adds it back to the zinc bath. This method reduces the amount of zinc in the waste treatment unit, therefore reducing the amount of metal that must be treated and disposed.

Figure 2. Conventional Clarifying, Precipitating Waste Treatment and Zinc Process Line



The conventional waste treatment process is illustrated in Figure 3. Zinc hydroxide is processed through a filter press and a dryer before being disposed. The wet volume of the zinc hydroxide can be reduced 4 to 5 times with this process, which results in lower freight and disposal charges as less volume (i.e., less water) would be shipped and disposed.

Figure 3. Waste Treatment (Clarifying Precipitation) Process



The RO zinc recovery process is completely separated from the clarifying, precipitating waste treatment system, the latter being common in metal plating operations. When Global Plating uses the precipitation system, the resulting sludge is sent for metals recovery rather than disposed as hazardous.

Cross Segment Uses

The RO process is applicable to a variety of plating operations, such as for bright nickel, where metals recovery is desired.

PROCEDURE

DEMONSTRATION

Following installation of the RO system as shown in Figure 1, trials were conducted during normal production on the zinc plating line. The plating process was not changed. The only alterations to the plating line were the piping to the RO system and to the atmospheric evaporator.

The atmospheric evaporator was located beside the zinc plating tank. Zinc plating solution was pumped to the evaporator where it passed through a series of large baffles. Air was blown down through the baffles, evaporating the water. The evaporation rate depended on:

- Air volume
- Air temperature
- Zinc plating solution temperature
- Zinc plating solution volume
- Baffle surface area

The atmospheric evaporator must evaporate enough zinc plating solution to accommodate the RO concentrate returning from the RO system. Without sufficient evaporation, the RO concentrate automatically diverts to the UF collection sump tank. When this sump is full, the RO concentrate can be diverted to the pretreatment storage tank. When these are full, the RO concentrate is automatically overflowed to the clarifying, precipitating waste treatment system where the zinc is precipitated into sludge.

The RO system operates as follows:

- 1. City water enters a water softener to remove any water hardness from the make-up water to the RO system (i.e., all make-up water should be de-ionized or RO water).
- 2. Zinc rinse water from the #1 zinc rinse tank flows to the UF collection sump tank at 2 gallons per minute.
- 3. This water is pumped by a 0.5 horsepower pump through the 5 micron filter cartridge, through the UF membrane, and collected in the pretreat storage tank.

- 4. The water is pumped by a 5 horsepower pump at 20 gallons per minute at 200 pounds per square inch through the RO membranes.
- 5. Water molecules pass through the membrane and return at 2 gallons per minute to the #2 zinc rinse tank. Because RO membranes are not completely efficient, approximately 20 percent of the chemicals leak through the membranes and return to the #2 zinc rinse tank.
- 6. The rejected chemicals from the RO membranes are directed back to the pretreat storage tank for another pass through the RO membranes. This sequence is followed until solution conductivity levels trigger the opening of a solenoid valve. The concentrated reject solution is sent to the zinc plating tank at 1 gallon per minute. The conductivity controller is set at 2,000 parts per million. The solution returning to the zinc plating bath is concentrated about 4 to 6 times.

Zinc Plating Tank Adjustments

Global Plating uses an acid chloride zinc solution with the following characteristics:

Zinc chloride: 4 to 6 ounces per gallon

Potassium chloride: 17 to 21 ounces per gallon

Boric acid: 3.5 to 5 ounces per gallon
Brightener: 0.05 to 0.1 percent by volume

Carrier: 3 to 4 ounces by volume

• pH: 4.5 to 5.5

Temperature: 65 to 90°F

This type of solution has been standard for approximately 20 years, and has been a replacement for zinc cyanide solution.

The acid chloride zinc solution must be run with organic additives that modify the crystalline structure of the plated deposit to improve its quality. The brightener can also improve deposit quality, but must be enhanced by a carrier (detergent), as the brightener is not water soluble. However, the carrier foams when agitated and with temperature increases. In the atmospheric evaporator, agitation of the zinc plating solution can send foam up the exhaust stack and onto the roof. Changing from one vendor's carrier to a potentially less-foaming one can take months of production, as the carrier is only removed by dragout of the zinc plating solution.

Temperature increases also affect brightener consumption and plating quality. Brighteners degrade in the plating solution is above 90°F. At such temperatures, plating in recessed areas of a part deteriorates.

Plating Trial With Reverse Osmosis Membranes

In conducting the RO membrane plating trials, Global Plating used the following procedure.

1. Zinc rinse tanks were dumped and made up with new RO water.

- 2. The atmospheric evaporator and the RO system were activated.
- 3. Samples of the RO reject, RO elutant, #1 rinse tank, and #2 rinse tank were analyzed for zinc, chloride, and conductivity. Zinc and chloride were analyzed by standard volumetric tests. Conductivity was measured with a conductivity meter.

RO membranes normally operate at about 80 percent efficiency due to inherent characteristic design and manufacturing parameters. When the reject and elutant are analyzed, the ratio between the two levels indicates efficiency separation. The efficiency expressed by this ratio also can indicate when the membranes are becoming clogged and need to be cleaned. As the RO and UF membranes become clogged, the conductivity ratio of concentrate to elutant and the conductivity ratio of the RO concentrate to the concentration in the #1 rinse tank reduce.

COST OF DEMONSTRATION

The total cost of the demonstration was \$42,548.42. EPA provided \$15,180 through the Pollution Prevention By and For Small Business Grant Program, and Global Plating contributed the balance of \$27,368.42.

RESULTS AND DISCUSSION

PERFORMANCE RESULTS

The ratio of RO concentrate to RO elutant started at 17:1 and remained above 10:1 for over one month, as shown in Table 1.

TABLE 1. RO Conductivity Ratios

Date	RO Concentrate Conductivity (ppm)	RO Elutant Conductivity (ppm)	Ratio Concentrate:Elut ant
9-29-92	1,500	20	75:1
9-30-92	1,700	100	17:1
10-1-92	2,000	150	13.3:1
10-7-92	1,400	100	14:1
10-9-92	1,400	100	14:1
10-23-92	2,800	200	14:1
10-26-92	1,900	100	19:1
11-10-92	5,100	500	10.2:1
11-12-92	3,400	500	6.8:1

11-13-92	3,800	500	7.6:1
11-17-92	5,000	600	8.3:1
11-30-92	2,500	200	13.5:1
12-14-92	1,500	300	5:1
12-21-92	1,500	300	5:1
12-23-92	1,100	400	2.7:1
1-5-93	1,400	400	3.5:1
1-8-93	1,300	300	4.3:1
1-13-93	1,500	400	3.7:1
1-14-93	1,300	500	2.6:1

A drop in the ratio indicated that the membranes were becoming clogged and were in need of cleaning.

Unfortunately, the atmospheric evaporator could not function efficiently, because the rainy California weather resulted in 100 percent humidity with 35° to 50°F temperatures in the plating shop. Without the evaporator, the zinc tank could not accommodate the RO concentrate.

Global Plating unsuccessfully attempted to "help" the evaporator by raising the temperature of the zinc bath to 100°F. At this temperature, foam (from the brightener in the zinc bath) came from the evaporator and moved up the exhaust stack. Experimentation with three different brighteners and carriers also resulted in unacceptable foaming. In addition, at 100°F, the brighteners were not reaching the recessed areas of the plated parts, thus lowering their quality. The zinc bath temperature was lowered to its normal operating temperature. Thus, the RO concentrate could not be added to the zinc tank and was instead sent to the waste treatment system and made into sludge (which was subsequently sent for off-site metals reclamation). No metals or chemicals could be reclaimed on site, nor could any costs be saved.

Another challenge concerned the cleaners used for the RO and UF membranes. These specialized cleaners are highly chelated, and when discharged to the waste treatment system, keep metals from precipitating in the clarifying sections. The waste treatment system then must be stopped and treated with sodium di-methyl dithiocarbonate to break the chelated bond. 300 pounds of this chemical (at \$0.87 per pound) were required for treatment.

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Incentives

RO and UF membranes are effective in capturing zinc metals for recycling.

Plating baths that operate at room temperatures use brighteners that are destroyed at the high temperatures required for conventional evaporators. A vacuum still, operating at 90 to 100°F, would be effective for the evaporation of zinc chloride baths.

Zinc and nickel platers may find the process useful. Nickel platers would not require the evaporation step, as nickel plating tanks, operating at 140° to 155°F, lose a great deal of water each day through evaporation. Other types of plating solutions could be recovered using RO membranes providing they met membrane compatibility requirements.

Barriers

The atmospheric evaporator did not operate due to the high humidity during the fall and winter months of 1992-1993. The evaporator functions well in the summer months (or in a dry climate). Because of this failure, Global Plating could not return any concentrated zinc rinse solution to the zinc plating tanks, nor could they demonstrate any cost savings.

The brighteners foamed in the evaporator when the temperature approached 100°F. Research must be conducted to select a zinc brightener that can be used at this higher temperature.

As a potential solution to the evaporator malfunction, a RO and UF membrane system could be placed in the zinc plating solution for water removal. The membranes would have to be tested to ensure they would function with the highly concentrated chemicals in the plating bath.

IN-GROUND PLASTIC CONTAINER PRODUCTION SYSTEM TO REDUCE NITRATE AND PHOSPHATE POLLUTION

by

Carl E. Whitcomb, Ph.D. Lacebark, Inc. Stillwater, OK 74076

ABSTRACT

Lacebark, Inc. investigated the use of a new in-ground, plastic container system that reduced nitrate and phosphate pollution from above-ground container nurseries. Such nurseries may use in excess of 2,000 pounds of nitrogen per acre per year; much of the fertilizer applied to containers may be lost through leaching and spillage. The technique proposed by Lacebark suggested that fertilizer rate could be reduced by 50 percent or more (compared to conventional above-ground containers) when plants are grown in plastic containers that are submerged in the earth. In addition, irrigation water demand could be reduced.

INTRODUCTION

PROJECT DESCRIPTION

Outline of Process/Product

The in-ground plastic container production system is a new concept that may reduce nitrate and phosphate pollution from container nurseries. Container nurseries may use over 2,000 pounds of nitrogen per acre per year compared to 100 to 200 pounds of nitrogen for conventional agriculture. Further, because conventional containers are above ground, they may tip or may be blown over, resulting in spillage of the soil and fertilizer and possibly damaging the plant. The temperature in an above-ground container may reach 100°F on a sunny day, causing slow-release fertilizers to release more quickly, and damaging roots such that their capacity to absorb nutrients is greatly diminished. Once the fertilizer exits the above-ground container, it is no longer accessible to the plant and becomes a pollution problem in soil and water run-off.

With the in-ground plastic container system, the containers cannot blow over, as the containers are submerged in the ground up to the rim. Temperatures in the container are typical of soil temperatures for the geographic area (73° to 78°F for this study). The cooler soil temperatures in the in-ground container improve the root's nutrient absorption efficiency compared to above-ground containers, and with the unique container design, any nutrients that are leached from the container are still accessible by the secondary nutrient recovery root system.

Unique Product Features/Advantages

The purpose of the project was to assess the practicality of growing plants in specially-designed containers that are submerged in the soil. This technique may reduce nitrate and phosphate pollution from containers, especially in sandy soils

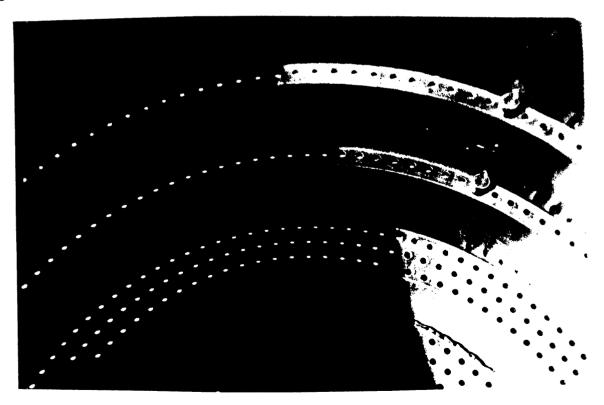
where capture and recirculation of run-off water is not feasible as a pollution prevention technique. Root zone temperatures in the in-ground container were at least 20°F cooler than in above-ground containers, thus improving root functions and avoiding root death due to the high heat.

The key element of the study was in-ground container that has over 1,200 openings, each 3/32-inches in diameter. Roots can "escape" through these holes to serve as a secondary nutrient recovery system; because of the small size of the openings and the strength of the plastic, the roots cannot expand. When the plant is harvested, the roots break at the point of constriction, which has little effect of the health and subsequent growth of the plant.

Product Drawing

Lacebark developed and arranged for the manufacture of the new plastic container design used in this project. The container is 12 inches in diameter and 10 inches deep, holding approximately 5 gallons of soil. The sides of the container are slightly tapered to permit easy removal from the injection molder. The container has 4 ledges on the sides, and each ledge and the container bottom contain over 1,200 "escape" openings, 3/32-inches in diameter. The container has no large drain holes as in conventional above-ground containers, as the large holes allow roots to escape and grow to considerable size, making harvesting difficult and severely shocking the plant if any roots must be severed. The in-ground container is shown in Figure 1. The pegs at the upper right aid in removal of the pot from the mold and prevent roots from circling within the container.

Figure 1. The In-ground Container



APPLICATION

Process or Products Replaced

The in-ground container can replace conventional above-ground containers that are subject to being blown or tipped over, resulting in fertilizer spillage and possible plant damage. In-ground containers promote lower root temperatures, decreasing the rate of fertilizer release and increasing the nutrient absorbing capacity of the root system.

Conventional field-grown nursery stock may also be grown in the in-ground containers, reducing fertilizer usage on a per acre basis. For example, when trees or shrubs are grown in field soil (without containers), the plant roots extend freely in all directions. Consequently, fertilizers are typically spread over the entire growing area, some of which does not contain plant roots. By growing plants in the field in inground containers, the fertilizers can be concentrated in the area where the roots develop. In addition, with the "escape" roots and the secondary nutrient recovery they provide, nutrient pollution will be minimized and crop response maximized.

Wastes Prevented

Fertilizer spillage from blown or tipped over containers is eliminated. Some slow-release fertilizers rapidly increase their rate of nutrient release as temperatures increase, as often seen in typical above-ground containers. With the in-ground containers, both plant roots and fertilizers are much cooler. This increases the capacity of the roots to absorb nutrients while reducing the rate of release of some slow-release fertilizers. The secondary "escape" root system that develops outside the in-ground container also may aid in reducing nutrient pollution in the soil and groundwater.

The higher cost of the in-ground container is offset by its reusability as a production tool. A plant can be grown in the in-ground container, harvested, and removed from the container when marketable size is reached. The in-ground container can be reinserted into the same cavity in the soil for reuse. The container in which the plant would be marketed would be a very thin, low-cost conventional plastic container or a paper mache container that is plantable by the end-customer. The development of a paper mache container to complement the in-ground container has distinct advantages by reducing the number of plastic pots destined for landfills.

The direct savings in fertilizer costs will be modest. By confining the fertilizer to a small area, there will be secondary benefits of reduced weed growth problems in adjacent areas.

PROCEDURE

DEMONSTRATION

Three nursery sites were selected in central Florida for the demonstration. Cherry Lake Tree Farm in Lake County, west of Orlando, is on the deep, rolling, sand hills generally referred to as the central Florida Ridge. The soils on this site are very low in organic matter averaging 1.1 percent, and have a consistency of ball bearings.

Water and nutrient retention is quite low, and the first water table is at a depth of approximately 30 feet, as estimated by the nursery manager.

Loyd and Ring Tree Farm and Holly Lane Tree Farm are in eastern Hillsboro County, approximately 20 miles east of Tampa, Florida. Both sites are flat with a shallow water table at a depth of approximately four feet. The soils on both sites are sandy, but the particle size of the sand is much finer in texture, with 3 percent organic matter. Water and nutrient holding capacity of these soils is substantially higher than at Cherry Lake.

Soil samples were taken from all sites before the assessment began. The analyses of these samples serve as a bench mark of soil nutrient conditions on each site and are noted in Tables 4, 5, and 6.

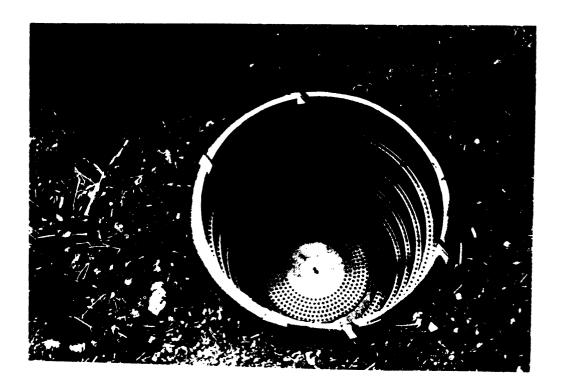
A tractor-mounted auger created the cavities in the soil into which the containers were inserted. Approximately 1.5 inches of the container remained above ground level. The container was filled with the existing sand field soil. Young trees were planted in the in-ground containers as well as directly in the ground (without containers) in a random pattern and watered. Subsequent waterings were by drip-irrigation. The plants were fertilized at a low, medium, or high rate according to fertilizer label directions. At Cherry Lake and Loyd and Ring Tree Farms, Osmocote 16-6-10 plus minor nutrients with an 8 to 9 month release rate was applied at a rate of 100, 200, or 300 grams per plant. At Holly Lane Tree Farm, Osmocote 14-14-14 with a 3 to 4 month release rate was applied at a rate of 50, 100, and 150 grams per plant.

Test plants at Cherry Lake Tree Farm were sweet gum, camphor tree, and Muskogee crapemyrtle. At Holly Lane Tree Farm, live oak, American holly, and loblolly pine were used as test plants. At Loyd and Ring Tree Farm, sycamore, citrus, and red maple were used as test species. All test plants were replicated four times with the three levels of fertilizer and with and without the presence of the in-ground plastic container. 216 trees were used in the study.

Soil cavities for the in-ground containers were dug using a 12-inch tractor-powered auger. A leg at the center of the auger functioned as a depth gauge.

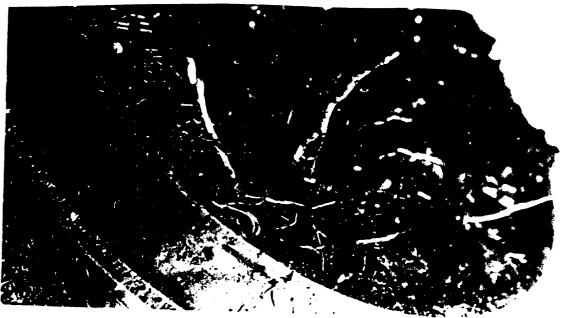
In Figure 2, the in-ground container is placed in the augered hole prior to filling with soil. The 3/32-inch holes may be seen, which allow roots to "escape" and function as secondary nutrient recovery systems outside the container. The four groves in the top of the pot hold the drip irrigation line in place to ensure accurate watering.

Figure 2. The In-ground Container in the Soil



In Figure 3, an in-ground container is removed from the soil after about 6 months. Note that the roots have grown through the small holes in the container.

Figure 3. In-ground Container After Removal from the Soil



EVALUATION PARAMETERS

The plants grew approximately four months at Holly Lane Tree Farm and for three months at the other two site prior to growth measurements and further testing for nitrates, phosphorus, and potassium. On June 13, 1993, height growth of all tree species at Cherry Lake and Loyd and Ring Tree Farms was determined. Height measurements were taken for the three test species at Holly Lane Tree Farm on June 17, 1993.

After measuring height growth of the trees, the plants in the in-ground containers were lifted from the earth and a soil sample was taken from directly beneath the bottom of the container. For trees grown in the soil without the in-ground container, a hole was dug beside the tree to a depth equal to or greater than the depth of the bottom of an in-ground container. A large metal spoon was bent to serve both as a depth gauge and to extract soil samples from beneath the plant at a location similar to that described for the in-ground container. 54 soil samples were taken and analyzed for nitrates, phosphates, and potassium using conventional agricultural soil test procedures.

After taking the soil samples, the containers were returned to the cavities and holes beside the non-container plants were refilled so as not to overly disturb the plants. The plants will be allowed to grow for another year to obtain long-term information.

ASSESSMENT SUMMARY

The roots of all species had grown sufficiently to extend through the 3/32-inch "escape" openings in the in-ground containers and into the soil below, and to a lesser degree, to the sides. The roots outside the containers absorb water and nutrients and send these to the leaves via the xylem, or central vascular strands, of the roots that remain intact through the small openings. The phloem, or outer root tissues, are squeezed out by the expanding xylem, thus the downward flow of sugars from the leaves in the phloem stops abruptly at the inner surface of the container. As a result, the roots outside the container are essentially nurse roots that capture water and nutrients that otherwise would have been lost from a conventional above-ground container, yet receive very few sugars from the leaves in return. Because of root constriction, the loss of these roots at a time of harvest has very little detrimental effect on the plant. Growth of the trees (height in inches) generally was best with the lowest fertilizer level or was approximately the same, regardless of fertilizer levels at all three test sites, as shown in Tables 1, 2, and 3. No significant differences in growth were noted in any tree species between fertilizer levels or presence or absence of the container.

TABLE 1. Plant Growth (Height in Inches) at Holly Lane Tree Farm, Plant City, FL

	FERTILIZER LEVEL					
SPECIES	LOW	MEDIUM	HIGH			
Live Oak						
Container	9.6	10.3	9.1*			
No container	10.3	11.6	12.1			
American Holly						
Container	7.0	6.3	5.3			
No container	5.7	5.3	4.7			
Loblolly Pine						
Container	24.5	24.0	20.5			
No container	27.9	24.7	25.7			

^{*} mean of four replications

TABLE 2. Plant Growth (Height in Inches) at Cherry Lake Tree Farm, Groveland, FL

	FERTILIZER LEVEL					
SPECIES	LOW	MEDIUM	HIGH			
Sweet Gum						
Container	7.7	6.1	4.5*			
No container	6.3	4.5	4.2			
Camphor Tree						
Container	4.6	3.5	3.0			
No container	4.8	4.3	2.2			
Muskogee Crapemyrtle						
Container	8.0	7.5	6.2			
No container	7.7	4.5	3.5			

* mean of four replications

TABLE 3. Plant Growth (Height in Inches) at Loyd and Ring Tree Farm, Plant City, FL

	FERTILIZER LEVEL						
SPECIES	LOW	MEDIUM	HIGH				
Sycamore							
Container	11.5	9.2	9.6*				
No container	11.5	11.6	12.6				
Citrus							
Container	7.6	4.9	4.3				
No container	6.1	6.1	4.2				
Red Maple, second t	flush of growth						
Container	8.25	6.5	4.7				
No container	7.2	4.9	4.0				
Red Maple, total gro	wth (first and second	flushes)					
Container	11.6	15.0	10.0				
No container	11.0	14.0	11.5				

^{*} mean of four replications

COST OF DEMONSTRATION

The total cost of the demonstration was \$33,072.85, with EPA providing \$16,002.17 through the Pollution Prevention By and For Small Business Grant Program, and the balance of \$20,852.85 contributed by Lacebark.

RESULTS AND DISCUSSION

PERFORMANCE RESULTS

The in-ground container worked well at Loyd and Ring Tree Farm where the soils were finer and had more organic matter, and results were similar to a preliminary study conducted in Oklahoma in 1991. At the Cherry Lake Tree Farm site with "ball bearing" soil, some benefits have resulted from reduced water movement through the soil during heavy rains. Benefits from Cherry Lake were less clear.

PRODUCT QUALITY VARIANCE

The quality and consistency of the in-ground containers were excellent once the initial plastics fabrication problems were solved. The variation in performance appears to be related to the soils and drainage conditions on the site rather than the product.

CONDITIONS THAT IMPACT PERFORMANCE

Clearly the Cherry Lake site was an extreme in terms of low soil organic matter and extremely coarse, deep sand, yet there are hundreds of thousands of acres of similar soil in central Florida. The results suggest that this technique performs better on sandy soils that have greater organic matter and water-holding capacities. Much of the sand soils of New Jersey, South Carolina, North Florida, and eastern Texas are similar to the Loyd and Ring Tree Farm site where the container performed as anticipated.

TABULATION OF DATA

Soil test results were compared for sites with and without the in-ground container for each of the three nutrient elements: nitrates, phosphorus, and potassium. The pre-test nutrient levels in the soil, both range and average, are noted to the right of the following tables.

Soil test results from Holly Lane Tree Farm (Table 4) show no significant differences among nitrates, phosphorus, or potassium with or without the in-ground container at any fertilizer rate. The levels of nutrients in all cases were similar to or lower than the soil test levels from before the treatments were applied. Soil nutrient levels were lower three months after the fertilizer was applied, suggesting that the slow-release fertilizer failed to release any appreciable nutrients. This problem is known to occur from time to time, as consistency in the time-release coating thickness of the fertilizer can be difficult to maintain.

TABLE 4. Holly Lane Tree Farm - Soil Nutrient Levels below the Pot Depth after approximately Four Months

Nutrient	t Nitrate (ppm)			Phos	Phosphorus (ppm)			Potassium (ppm)		
Fertilizer Level	Low	Mediu m	Hig h	Low	Mediu m	High	Low	Mediu m	High	
Container	3 5 4	3 1 4	5 4 3	341 326 341	329 372 338	326 326 341	16 15 19	16 30 13	14 15 21	
Mean	4.0*	3.0*	4.0	336*	346*	331*	16.7*	19.7*	16.7 *	

No container	1 1 3	3 5 4	2 4 2	332 335 355	341 390 372	343 381 346	15 · 21 15	13 28 33	14 79 18
Mean	1.7*	4.0*	2.7*	340*	368*	353*	17.0*	24.7*	37.0 *
Baseline readings (pre- study)		ange 8- an 8.8 p			nge 350-3 ean 364 p			ange 29-7 an 49.8 p	

^{*} Not significant

At Cherry Lake Tree Farm, nitrate levels in the soil below the bottom of the container were not significantly different for the container versus no container at the low and medium fertilizer levels. However, at high fertilizer application levels, high levels of nitrates were found below the in-ground container (24.0 ppm) versus a comparable depth below the trees with no container (8.3 ppm). Phosphorus levels in the soil below the bottom of the containers also were not significantly different for the container versus no container at all three fertilizer levels. Potassium levels in the soil below the bottom of the containers were not significantly different for the container versus no container at the low and medium fertilizer levels. A significant difference was seen in the high-rate application of potassium beneath the container (25.7 ppm) versus no container (7.7 ppm). Data for Cherry Lake Tree Farm may be found in Table 5.

TABLE 5. Cherry Lake Tree Farm - Soil Nutrient Levels below the Pot Depth after approximately Three Months

Nutrient	Nitrate (ppm)		Phos	Phosphorus (ppm)			Potassium (ppm)		
Fertilizer Level	Low	Mediu m	High	Low	Medi um	High	Low	Mediu m	High
Container	18 19 10	12 23 7	26 16 30	54 54 31	85 54 56	56 62 51	20 21 13	19 48 21	38 23 16
Mean	15.7*	14.0*	24.0 †	49.7*	65.0*	56.3*	18.0*	29.3*	25.7 †
No container	5 18 8	35 9 7	9 4 12	36 51 47	62 49 51	44 46 48	5 22 10	45 18 13	8 8 9
Mean	10.3*	17.7*	8.3†	44.7*	54.0*	46.0*	12.3*	24.0*	7.7†

Baseline readings (pre- study)	Range 5-13 Mean 9.3 ppm	Range 36-53 Mean 45.2 ppm	Range 8-21 Mean 11.2 ppm
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^{*} Not significant

At Loyd and Ring Tree Farm, nitrate levels in the soil below the bottom of the container were significantly lower beneath the in-ground container versus no container for the low and medium fertilizer level. No significant difference was seen at the high fertilizer level. Phosphorus levels and potassium levels (at all three fertilizer levels) were also not significantly different in the soils beneath the container versus no container. Data from Loyd and Ring Tree Farm are shown in Table 6.

TABLE 6. Loyd and Ring Tree Farm - Soil Nutrient Levels below the Pot Depth after approximately Three Months

Nutrient	Nit	Nitrate (ppm)		Phos	Phosphorus (ppm)			Potassium (ppm)		
Fertilizer Level	Low	Medi um	High	Low	Medi um	High	Low	Medi um	High	
Container	8 6 9	8 6 12	10 7 17	40 33 56	91 26 68	47 39 83	6 11 7	12 6 9	9 12 21	
Mean	7.7†	8.7†	11.3*	43.0*	61.7 *	56.3*	8.0*	9.0*	14.0 *	
No container	33 12 11	21 9 17	11 5 21	60 24 48	41 25 38	22 13 69	47 9 11	17 6 16	11 5 33	
Mean	18.6†	15.7 †	12.3*	44.0*	34.7	34.7*	22.3*	9.7*	16.3	
Baseline readings (pre- study)		nge 11- n 17.3 p			nge 38- n 38.6 p		Ra Mea	nge 59- n 65.3 p	79 pm	

Not significant

Conclusions

A comparison of soil nitrate levels at a depth just below the bottom of the container for Cherry Lake Tree Farm versus Loyd and Ring Tree Farm probably

[†] Significant at 5% level

[†] Significant at 5% level

reflects the differences in soils between the two sites. The soil at Cherry Lake Tree Farm was deep, "ball bearing-like" sand with an estimated depth of 30 feet to a water table. The organic matter content averaged 1.1 percent. By contrast, Loyd and Ring Tree Farm had a finer sand soil with much better water-holding characteristics and an estimated depth to a water table of four feet. The organic matter content averaged 3.0 percent.

With the extremely well-drained sand at Cherry Lake Tree Farm and the heavy rains that occurred during the period of the study, the container may have retarded downward water movement and leaching compared to where no container was present. By contrast, at Loyd and Ring Tree Farm that had a higher water table, and where soils were also sandy, but of much finer texture and higher organic matter, the benefits of the container fit the rough data of the preliminary study.

Phosphorus moves very little in soils, even in extremely coarse sand soils such as at the Cherry Lake site. The lack of difference with this nutrient between the container and no container trials was not unexpected.

Potassium moves at a modest rate under conditions of sand soils, low organic matter, and heavy rainfall. A restriction was noted in potassium movement at the Cherry Lake site similar to that observed for nitrates.

The temperature of the soil at the time of the second sampling two inches below the surface averaged 78°, 73°, and 73°F for the Cherry Lake, Loyd and Ring, and Holly Lane sites, respectively. On the same days while taking soil samples, the soil temperatures in above-ground containers averaged 112°, 109°, and 104°F at the same three sites, respectively. The temperature readings are the means of six measurements.

COST/BENEFIT ANALYSIS

Lacebark feels that because the in-ground container is so unique as compared to conventional procedures, and since a full-scale production trial has not been conducted, further cost/benefit estimates would be mostly fiction.

The unique in-ground containers performed well in terms of (1) reduced root zone temperatures and corresponding reduced fertilizer release rates; (2) reduced evaporation and water use; (3) avoided fertilizer loss, as trees could not be blown or tip over, even though some were more than six feet tall; and (4) reduced nitrate movement. Benefits from this unique in-ground container will likely increase with additional tree growth over time.

The cost of augering the holes to install the in-ground containers is one-time for a number of years of production. Whenever a tree is harvested, another container of the same design and size would be inserted, keeping the cavity open. The drip irrigation system is also a one-time cost that will function for many years. The two above items plus the cost of the in-ground containers are the primary up-front expenses.

The in-ground container, as currently manufactured with a single cavity mold, has a retail cost of \$1.60, as compared to a volume-manufactured, blow-molded

container that sells for \$0.51. Mass producing the container would reduce the production price drastically, lowering its selling price and improving its potential for acceptance in the marketplace. The container cost can be spread over an anticipated life of four to six uses or more.

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Incentives

The design of the container and the root "escape" system was satisfactory. Only minor modifications of the mold are required, specifically, a larger rib and more frequency around the inner circumference of the container to further reduce root circling.

Placing the in-ground containers in the soil solves three major problems commonly noted with above-ground containers for nurseries and tree farms: heat injury to roots in summer; cold injury to roots in winter; and blow over. The convenience of drip irrigation and resulting water savings are improvements over overhead sprinkler irrigation.

The target industry consists of the many nurseries on sandy soils currently producing plants in conventional above-ground containers. Good soil drainage may be a requirement for this technique. This system may not work on sites where water movement through heavier (such as clay) soils is slow. In-ground containers may also have appeal to non-container field nurseries with sandy soils, which do not have adhesion qualities for good digging with mechanical tree spades.

Additional assessments are planned for geographic areas where soils are appropriate for the use of in-ground containers. As with any new concept, successful demonstrations in similar growing conditions are usually necessary to promote technology adoption by nurseries and tree farm operators.

<u>Barriers</u>

Two major barriers are: (1) the initial cost of the container; and (2) in-ground containers are a new technology. Demonstrations in various geographic areas can be very effective in gaining familiarity with new technology. Constructing a mold such that four or more containers could be fabricated at one time versus the current single cavity mold would substantially reduce the unit cost to the end user.

REUSE OF METAL FABRICATION WASTEWATERS VIA A NOVEL ULTRASONIC COALESCENCE PROCESS

by

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ABSTRACT

Metal working and finishing operations generate wastewater containing cutting and cooling fluids along with metal particles and oils. Currently, this entire volume of wastewater must be disposed of as a hazardous waste. S.R. Taylor and Associates' (SRTA) project studied the effectiveness of a novel ultrasonic technique that separates the hazardous contaminants from the fluids so that the bulk microemulsion phase can be reused. Up to 90 percent volume reduction of wastewater from small metal fabricating shops was projected. SRTA built a multistage, multi-frequency operation to assess the commercial potential of the recovered oil phase and the solids sludge, and to evaluate the long-term stability and usefulness of recycled fluids.

INTRODUCTION

PROJECT DESCRIPTION

Typical metal working and finishing operations generate stable suspensions containing hazardous solid metal particles as well as oils in their cutting and cooling fluids. Currently, the entire volume of this wastewater must be handled and disposed of as hazardous waste. The only acceptable methods to separate these hazardous materials are expensive and inefficient, especially for small operations. The purpose of this project was to assess the effectiveness of a novel ultrasonic technique that achieves pollution prevention by separating the hazardous contaminants from the fluid so that the bulk microemulsion phase can be reused. Successful implementation of this process could feasibly lead to reuse of up to 90 percent of the volume of wastewater from small metal fabricating shops. This project included the construction of a full-scale unit for long-term quantitative and qualitative on-site evaluations of the equipment, process, and reused fluid.

Current metal cutting and finishing operations use skimming and filtration to separate the wastes from their water-based cutting fluids. The residual liquid from the filtration consists of ultrafine metal particles containing hazardous heavy metals such as lead, zinc, and cadmium, and a highly stable emulsified oil-in-water phase. Currently, the only accepted method of cleaning this waste mixture for disposal is distillation, leaving a hazardous metal sludge, but allowing for the water to be safely disposed. Distillation is expensive, both for initial capital investment and for operation. This expense deters many metal finishers who, instead, simply filter the fluids and attempt to recycle them. When recycling is no longer possible, the entire volume must be disposed of as hazardous waste. Other methods for separation of oil from

wastewater, such as air flotation, also have limited effectiveness. They are not suitable for separation of multiphase systems involving stable emulsions with suspended solids nor are they cost effective for small treatment volumes. In all of these cases, the separation and removal of the small oil droplets and metal particles is so difficult because the particle size is so small and becomes increasingly difficult as the particle size is reduced. For example, the oil/water emulsion leaving an American Petroleum Institute separator normally has oil droplets less than 30 microns in diameter and concentrations less than 200 milligrams per liter (Sylvester, N.D.and J.J. Byeseda, "Oil/Water Separation by Induced-Air Flotation," Soc. Pet. Eng. J., 20, p. 579, 1980). A separation process is needed that will be effective on small particle sizes in small concentrations.

The overall goal of this project was to further develop and optimize ultrasonically enhanced coalescence and separation of metal working fluid waste to allow reuse/recycling of the separated phases. The approach was to build a multistage, multi-frequency operation, to evaluate the commercial potential of the recovered oil phase and the solids sludge, and to conduct actual site tests to ascertain long-term stability and usefulness of the recycled fluids.

Theory

The theoretical basis for separating a suspension of fine particles involves the use of ultrasonic waves transmitted into the bulk fluid in a standing wave pattern. This causes small particles to collide with the large particles and thus coalesce to a size large enough to enable separation from the bulk fluid.

Two theories have been developed that treat the covibration of suspended particles in a fluid medium. The relative motion, R, between the particle and the medium, due to ultrasonic vibration, is a convenient means of expressing the phenomenon. The Brandt, Freund, and Hiedemann derivation (Brandt, O., et al., "Zur Theorie der Akustischen Koagulation," Kolloid-Zeitschrift, 77, 103, 1936) leads to nearly the same mathematical expression given in later work by St. Clair (St. Clair, H.W., Industrial Engineering Chemistry, 41, 2434, 1949).

The Hiedemann form is:

$$R = \frac{Xp}{Xm} = \frac{1}{\{1 + (\frac{\pi d^2 f(\rho_p - \rho_m)^2}{9\eta})\}^{1/2}}$$

where Xp, Xm = amplitudes of motion of particles and medium, respectively (cm) d = particle diameter (cm) f = frequency (Hz) = densities of particle and medium, respectively (g/cc) = viscosity of the medium (poise: g/cm/sec)

Calculations based on the Hiedemann equation (taking into account the density and viscosity of water to show the variance in R as a function and particle size) indicate that within the frequency range of 50 to 100 kilohertz, it should be possible to obtain a difference in the relative motion of solid particles and emulsified liquid droplets (5 to 50 micron size) as long as the density difference between the emulsified phase and the continuous phase is 0.1 grams per cubic centimeter or greater. Therefore, theoretically it is possible to coalesce oil and metal particles in a water-based medium.

Unique Process Features/Advantages

The unique feature of this process is its ability to allow the recycling of much of the fluid used in metalworking processes since it contains up to 90 percent water. The process can be operated as a simple, inexpensive service provided by outside vendors already working with disposal of these fluids. This reduces the cost to the consumer while maintaining close control on hazardous wastes in the manufacturing plant.

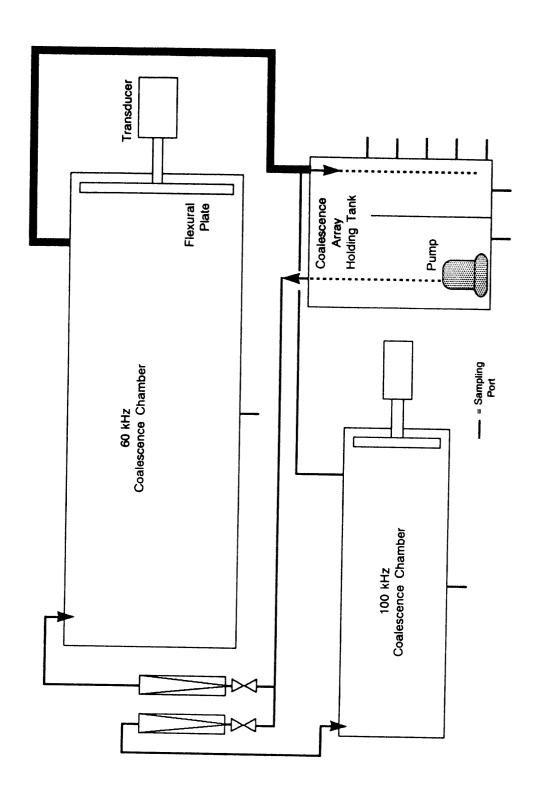
Previous research under a Small Business Innovative Research Grant verified the relationship of particle size density and frequency of operation. The results of these studies proved the "soluble oil"/water microemulsion is the bulk phase and the coalescence method effectively removes the excess oil and larger solids from this phase. This leaves a cleaned bulk phase that could be 100 percent reused.

Particles below 15 microns were not expected to be greatly influenced by the 60 kilohertz vibrations used in these earlier studies. Also, with oil present in the system, the viscosity increases slightly, and the oil may coat the suspended solids. At a higher frequency (100 kilohertz), solids separation should improve. However, much lower flow rates will be required to account for the higher attenuation at the higher frequency. Better solids rejection should be achieved by using a staged coalescence with two chambers, one at 60 kilohertz, and a second at 100 kilohertz. Manufacturing and testing of this multi-stage array were the main purposes of the SBIR project. In addition, a portion of the reconditioned fluids was returned to the operators to learn whether the cleaned fluid was suitable for reuse. Each sample from the earlier studies proved satisfactory for reuse and did not seem to go rancid as quickly. Verifying operator reuse was also part of this project.

Process Schematic

A schematic drawing of the process is shown in Figure 1. As can be seen, it is a simple, continuous flow system that allows any volume of fluid to be treated.

Figure 1. Schematic of Continuous Flow-through Array



The coalescence chamber contains a flexural plate developed by SRTA. When power is applied to the system, the plate radiates a sound beam in a circular area. The *axisymmetric* quality of the sound beam is very effective in causing the smaller particles in the liquid to collect together as larger particles, or *coalesce*. The larger particles then separate into different phases by gravity and differing densities.

When a stable suspension of metal cutting fluid waste is pumped through this coalescence chamber and the phases are allowed to separate, a water/"soluble oil" microemulsion remains between the oil and metal sludge phases. This microemulsion may be removed and reused as a metal cutting fluid. The top oil layer may also be removed as a machine-ways oil or recycled. The bottom metal sludge layer may be sold for metal values, or disposed of properly.

Implementation in Industry

Based on results from the process testing, specifications can be developed for an actual system. In the tested scenario, the system can be envisaged as a service operation much like the current parts cleaner service systems. A cooling fluid holding tank (50 to 100 gallons capacity) could be installed at the customer's facility. The coalescence equipment would be mounted on a portable skid. The service operator would visit the customer's facility, treating the cooling fluids, and pumping the treated fluids into the holding tank where settling would occur. As the customer needs cooling fluid, he removes it through taps on the side of the holding tank. Periodically, the separated top oil phase could be collected and either used internally at the facility or sold to an oil recycling firm. The sludge, which settles to the bottom of the holding tank, would need to be removed and sold for recovery of metals; as a final option, it must be disposed of as hazardous.

The cost for the proposed system is minimal since the only operating expense is for electricity. Capital costs are relatively low due to the simplicity of the system. To date, S.R. Taylor has successfully treated liquids at flow rates up to 10 gallons per minute. Large volume treatment will be handled using a modular approach with several units operating in parallel to provide the desired treatment rate. Also, large operators may prefer to have the equipment installed on site to allow more frequent operation than could be reasonably handled by a service company.

From the customer's point of view, no water is discharged, the valuable oil is recovered, and the hazardous sludge is handled by specialists. By reusing the bulk microemulsion phase, the oil and metal values are essentially concentrated. If this concentration is sufficient, these materials represent new resources rather than hazardous wastes. The overall goal is to reuse/recycle all materials contained in contaminated metal cutting fluids.

APPLICATION

Process/Products Replaced

Current practice requires that metalworking facilities either dispose of the used fluids as hazardous waste or treat the fluids on site to completely separate the oil, solids, and water phases. Even then, the oil and solids phases need to be handled as

hazardous wastes. Systems designed to provide such complete separation are expensive and not easy to implement for small operators.

Replacement of these expensive processes with a simple service that allows most of the cooling microemulsion to be recycled will greatly ease the burden of compliance on metalworking shops, particularly small operators.

Wastes Prevented

Industries that discharge their wastewaters directly to streams, rivers, and lakes have to comply with limits set by the U.S. Environmental Protection Agency and their state on the contaminant content of their effluents. Typical contaminants may include oils and grease; oxidation and biodegradation products; and colloidal particles of heavy metals. Such effluents come from such sources as refineries; de-inking operations; metal plating; iron and steel plants; chemical processing plants; barrel and drum cleaning; washrack and equipment maintenance; mill waste; and aluminum forming.

Of these industrial wastewaters, metal fabricating processes have some of the highest oil contamination concentrations as seen in Table 1. For this reason, these fluids have been selected for initial testing although the proposed method should have applicability to a large number of these wastewaters. Each year, approximately 260 to 800 million gallons of machining fluids are used in the U.S. When used, these "soluble oil" and water fluids tend to form stable suspensions with hazardous metal particles. Removal of the excess oil and solids from the suspension would allow the remaining water/"soluble oil" fluid to be reused.

TABLE 1. Typical Oil and Grease Contents

Source	Range of Oil/Grease Concentrations (percent)
Wastewater	
Sewage	0.001 - 0.01
Food Processing	0.01 - 0.1
Textile	0.001 - 0.005
Petroleum Refining	0.01 - 0.1
Primary Metals	
Rinse Waters	0.001 - 0.1
Concentrate	1 - 5
Metal Fabrication	1 - 15
Metal Cleaning	
Rinse Waters	0.001 - 0.1

Source: Kulowiec, J.J., Pollution Engineering, 11,49, 1979.

Cross Segment Uses

Other industries potentially benefitting from this separation technology include chemical processing for separation of liquid-liquid emulsions, the oil industry for cleanup of process waters from enhanced oil recovery operations and for the improvement of inertial separators, and food processing plants for wastewater treatment.

While the technology has application to a wide variety of separations, it is a pretreatment for use prior to other separation techniques, such as gravity settling. Hence, the technology can be usefully applied to any system that has two insoluble phases that have a sufficient specific gravity difference to allow the ultrasonic standing wave to set up relative motion differences between the particles. This leads to an increase in particle size distribution that allows easier separation.

PROCEDURE

DEMONSTRATION PROCEDURE

The following objectives were set as the next logical steps toward refining the coalescence separation process. Successful completion of these objectives should provide answers to the questions listed below.

- 1. Quantitatively evaluate effectiveness for providing a usable fluid over extended periods.
 - How long (i.e., how many cycles) can the emulsion be reused? What percentage of the material is reusable following a cycle? Is this percentage a function of time/number of cycles? What other variables may affect this percentage?
- 2. Qualitatively evaluate process effectiveness including determining seasonal (summer versus winter) effects and obtaining operator feedback on reused fluid.
 - Operators have complained that cooling fluid becomes rancid during summer months. In previous testing, metal operators noted that the rancid odor disappeared after separation. Will this continue to be the case after continued reuse? How does the reused fluid compare to new fluid? Does anything need to be added to the emulsion before reuse?
- 3. Determine economic advantages for a metal operator using this process.
 - What are the commercial values of the oil and metal sludge layers? Will the oil need to be recycled or can the separated oil be used as is? Can the metal sludge be sold as a resource for metal values, or will it need to be handled as a hazardous waste?

The following work plan was proposed to meet the above objectives.

1. Construct portable multistage, multi-frequency array. The objective of this task was to construct a multistage, multi-frequency system to enhance the rate of separation by incorporating quiescent zones into the pump-around loop. Based on prior testing, improved solids coalescence can be achieved by operating at a higher frequency up to 100 kilohertz. However, increasing attenuation effects limit the flow rate, thereby limiting the treatment rate at these higher frequencies. The proposed solution was to conduct a simultaneous multi-frequency operation. This design allowed operation at rates up to 10 gallons per minute while treating a portion of the stream at 100 kilohertz at a flow rate of 2 to 3 gallons per minute. After assembly, the system was tested with simulated fluids to verify proper operation.

Since both streams are recombined in the holding tank, the entire volume eventually passes through both treatment chambers as long as the fluid is treated for a sufficient period to allow several passes through the slower chamber.

Ceramic crystals from Piezo Kinetics, Inc. were used to build the 60 and 100 kilohertz transducers for the two treatment chambers in the multistage array.

A magnetic-drive chemical pump with a Ryton pump housing was purchased for the portable array from Little Giant Pump Company. The pump handles a maximum pressure of 12.7 pounds per square inch at a maximum rate of 20.0 gallons per minute. The pump uses a one-eighth horsepower engine, however actual energy consumption by the pump and the entire array was measured throughout the testing.

The completed multistage array was mounted on a wheeled skid for ease in transportation.

2. Install holding tank on site. The on-site service array consisted of a 50-gallon separation tank that was installed at Delta Manufacturing, a stainless steel fabricator. The tank was made from plexiglass to allow observation of fluid separation, and several taps were located along the side of the tank to permit removal of "cleaned" fluid for reuse. The tank also included a tap on the bottom for periodic sludge removal. The tank was constructed with a removable top to allow for adequate cleaning prior to removal from the test site.

Delta chose to use the tank for storage of fluids from a grinder and a saw. The resulting 20 gallons of dark grey fluid constituted the test sample. This material normally would be disposed at this stage of contamination. Delta planned to replace this fluid with fresher solutions from a lathe; however, this did not occur in the time allotted for the testing, so the initial dirty sample was the only sample treated.

3. Treat suspension at regular intervals through varying weather conditions. On site testing required periodic visits to treat the fluids to promote settling and separation of the individual phases. The schedule called for treatments every two weeks throughout the year. The actual schedule is provided in a later section.

- 4. Interview metal operators using recycled fluid. The test site operators were interviewed to obtain an evaluation of the fluid after long periods of recycle (i.e., how does the fluid respond after 2 recycles?, 5 recycles?).
- 5. Perform composition tests of the clarified water layer. The goal of this task was to determine the level of oil, grease, and total suspended solids (TSS) in the water phase for recycle. These analyses were done by an outside testing laboratory after all test samples had been taken. Oil and grease content was measured using EPA method 413.1 and TSS was measured using EPA method 160.2.

EVALUATION PARAMETERS

SRTA designed a storage tank, which was built by General Plastic Fabricators of Tulsa, Oklahoma, for on-site installation. The tank was fabricated from transparent acrylic for maximum visibility of the fluids throughout the testing process. The tank was 4 feet, 8 inches from the top to the base plate, 12 inches wide, and 18 inches deep, for a volume of approximately 50 gallons. The top and base of the tank were flanged 1 inch on all four sides and are connected with a series of bolts to allow removal for easy cleaning. Two holes were drilled in the top plate for insertion of thermorneters to monitor temperature changes from treatment to treatment, and before and after treatment. The base plate was molded into a sloping funnel shape with a central 2 inch plug for removal of metal sludge.

The tank was filled with water and checked for leaks after 12 hours. No moisture was noted at any joints or around the base of the tank. Due to the weight of the water and the pressure exerted on the walls of the tank, supports were added on all four sides of the tank, 10 inches and 24 inches from the base plate, for extra strength.

On the side of the tank, six, one-half-inch diameter spouts were placed 9 inches apart, starting 2 inches from the base plate continuing to 6 inches from the top. A 3-inch wide strip of acrylic was placed under the spouts for reinforcement. These spouts were for removal of selected layers of the separated fluids.

Delta received the appropriate soluble oil for use with the grinder, and put a sample of the wastewater from the grinder sump into the holding tank in late October 1992. This represented the worst fluids generated by Delta, as the material was ready to be disposed of; no other method was used to try to recycle this fluid.

Delta continuously added used fluids through the top plate. Every two weeks, the fluids were pumped from the tank through a hose connected 8 inches from the base plate. The fluids were pumped through the multistage, multi-frequency array into the coalescence unit; they were returned to the tank through the top plate to settle into layers.

The water was very dirty with a dark grey color, and had apparently decomposed significantly. SRTA advised Delta that it was unlikely that the treatment could clarify this wastewater. Delta was more concerned that the fluid could be

cleaned sufficiently for reuse. (As noted below, this became an issue of user opinion rather than analysis.)

TESTING

The sample liquid was prepared for testing as described above. Additional fluids were added to the sample midway through the program. This waste sample was treated over three months as listed in Table 2.

TABLE 2. Actual Schedule for Coalescence Treatment

Treatment #	Cumulative Time, weeks	Date
1	0	November 6, 1992
2	2	November 16, 1992
3	3	November 25, 1992
4	5	December 9, 1992
5	8	December 29, 1992
6	11	January 25, 1993
7	12	February 4, 1993

Testing consisted of attaching the treatment array to the holding tank, pumping the fluids out of the tank, through the two treatment chambers, and back into the tank in a continuous flow cycle. The ultrasonic power generator activated the plate(s). The initial treatment on November 6, 1992 was done at 60 kilohertz only; the 100 kilohertz plate was not operational due to an electronic failure in the power generator. All subsequent tests were conducted with both plates activated. The individual test dates are described below.

- November 6, 1992: This initial test verified that the fluids could be pumped through the treatment array and verified that the 60 kilohertz plate was running. Samples were taken before and immediately after treatment (approximately 30 minutes). The fluid has a fairly strong, rancid odor.
- November 16, 1992: No fluids were added or removed from the tank. Treatment was the same as the first test. Two samples were taken: one prior to treatment and one 30 minutes after treatment. The return water stream, which entered the holding tank vertically through the top plate, caused significant agitation of the remaining fluid in the holding tank. Since this could cause resuspension of already-settled solids, this was modified prior to the remaining tests.
- November 25, 1992: No fluids were added or removed from the tank.
 Treatment was similar, but the return line to the holding tank was repositioned

to allow the water to flow in horizontally before dropping into the remainder of the water in the tank. This greatly reduced the agitation of the water. Two samples were taken: one prior to treatment and one 30 minutes after treatment. The oil and solids content had been reduced. However, a rather large algal/bacterial growth had formed on the top oil layer. These cultures exist in all shops and are a serious deterrent to fluid reuse. The fouled fluid has a noxious odor, and was separated prior to the next test. The water itself does not have a bad odor and, after treatment, has the smell of oil, not the bacterial byproducts. The color of the fluid is still a dark grey. This color, along with the growth on the oil layer, has inhibited the personnel from recycling the water phase.

- December 9, 1992: A skimmer was used to remove the top oil layer that had significant algal and bacterial growth and a foul odor. No fluids had been added or removed. The need to recycle some of the fluid was discussed with Delta personnel. Again, two samples were taken: one prior to treatment and one 30 minutes after treatment.
- December 29, 1992: Two gallons of additional fluid from the grinder had been added to the tank, although none of the fluid had been recycled. Again, two samples were taken: one prior to treatment and one 30 minutes after treatment. Visual observation of the two samples showed the separation easily. The treated sample was much lighter in color and clear separation was seen. However, even with the enhanced separation, the fluid remained a dirty color, and this appeared to inhibit personnel from recycling the water phase even though it was obviously cleaner than the starting material.
- January 25, 1993: Delta planned to recycle some of the fluid before the next treatment. After removing several gallons of the water phase, the odor was determined to be too strong to allow recycling. SRTA believes that the liquid was allowed to be stored too long and should have been recycled much sooner. This delay permitted bacteria and algal cultures to overwhelm the fluid.

The fluid was treated for the last time and sampled again. Although the water still smelled strongly at times, the operators did agree that it was no worse than the initial material. This means that the treatment was effective in controlling the fluid composition even though this particular sample was too fouled at the start.

• February 4, 1993: Delta indicated that they would replace the fluid with material from a lathe, but this had not yet been done. (The lathe fluid never gets as dirty as the grinder fluid, so it has a higher potential for recycling.) One final sample was taken.

ASSESSMENT SUMMARY

Delta Manufacturing did not recycle any of the sample nor did they fulfill their promise to provide a fresher sample. This limits the usefulness of these tests. However, future development can proceed quickly, as SRTA has a complete, mobile system that can be applied for further testing.

Although the specific goals of this project were not fully accomplished due to problems with the availability of test samples, SRTA has achieved a significant step towards commercialization by providing the first long-term data and by providing a system for additional testing.

As a result of having conducted this project, SRTA presented the results at several conferences including the EPA and DOE Waste Stream Minimization and Utilization Technology Fair, April 22-23, 1993, Austin, Texas.

RESULTS AND DISCUSSION

PERFORMANCE RESULTS

Technically, the system operated correctly and SRTA obtained similar separation results to those observed in earlier studies. Unfortunately, the actual sample used for these tests was an extremely contaminated sample and even operation at 100 kilohertz did not product sufficient visual separation to satisfy the user, although the analytical results were very good. Table 3 shows a comparison of the analytical results from an earlier sample and the current sample.

TABLE 3. Ultrasonic Coalescence Data for the Actual Cooling Fluid Samples

	Oil & Grease, mg/L	% Decrease	TSS, mg/L	% Decrease
Sample A - Prior Study				
Initial mixed sample	26,456		757	
Ultrasonic, middle layer	17,656	33	551	27
Sample B - Current Sam	ple			
Initial mixed sample	27,800		3,430	
Ultrasonic, middle layer	10,200	61	980	72

The first difference in these two samples is that Sample B (in the current study) had a much larger TSS than the earlier sample. As a result, although both the oil and grease and TSS levels were greatly reduced, the TSS still was larger than in the earlier sample. However, the earlier sample was entirely suitable for recycling. Although oil, grease, and TSS content were reduced sufficiently to allow successful recycling, the dark color of the treated fluid (presumably resulting from the high initial solids content) was an impediment to user comfort. Thus, none of the fluid in the current study was actually recycled.

Clearly, the process has some limitations on the quality of fluid that can be accepted for treatment. If the fluids are badly contaminated at the start, even good analytical results will not convince the user to recycle the fluid.

PRODUCT QUALITY VARIANCE

While separation was better than that achieved in earlier tests, results for TSS were somewhat disappointing. SRTA had anticipated greater solids rejection with the addition of the 100 kilohertz chamber. Two reasons are suspected for this product quality variance.

First, the starting fluid that was taken from a grinder sump tank was already at the point at which the operator would normally discard it. The fluid had already reached a level of deterioration that would not normally be considered "cleanable" as seen by the very dark color and the high TSS of the suspension prior to treatment. This material probably had a high concentration of extremely fine particles, and such fine material may not have been easily coalesced even at 100 kilohertz.

Second, although SRTA tested the system at both 60 and 100 kilohertz before using it to treat the fluids, a problem was discovered with the power generator after completion of the first two treatments: the 100 kilohertz section may not have been fully optimized. SRTA made modifications to the system that improved its performance, but this may have affected the initial results.

CONDITIONS THAT IMPACT PERFORMANCE

Clearly, the higher the solids loading, the poorer the performance of the system. These microemulsions can act much like thixotropic fluids to stabilize suspended solid particulate. Further testing with the 100 kilohertz section alone should be conducted to determine maximum tolerable solids content and to further optimize the system to treat high solids content fluids.

TABULATION OF DATA

Testing Observations

The fluid was so dirty at the beginning of the test that it represented a worst-case test. Since the treatment was not intended to provide a complete separation, the resulting fluid, although clean enough to recycle, was not suitable to the users' subjective judgement. This is an important factor leading to commercialization. Analytical characterization can verify separation and improve user confidence in recycling. The first requirement for the user would be to start with reasonably fresh fluids. The process then would operate to maintain fluid cleanliness.

The basic treatment procedure was simple to conduct. Hookup required simple connection to two hoses, and a single 110 volt outlet can provide power. All treatments lasted 30 minutes. Total time required to connect the system, treat, and clean up was less than one hour.

Analytical Results

Analytical results for oil and grease analysis and TSS are listed in Table 4 and are shown graphically in Figure 2. The fluid composition was greatly affected by the treatment. Reductions in oil and grease content reached 61 percent, while 72 percent

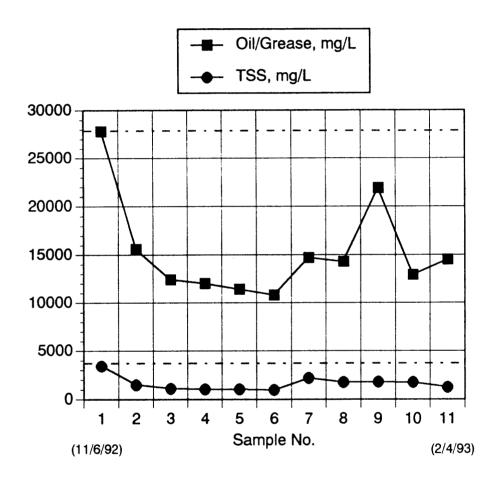
reduction in TSS was achieved. These results were better than those obtained in the earlier studies on cleaner feed stocks.

TABLE 4. Analytical Results for Treatment Samples

Treatment Number	Oil & Grease, mg/L	TSS, mg/L
1 (initial sample)	27,800	3,430
2	15,600	1,500
3	12,400	1,120
4	12,000	1,050
5	11,400	1,050
6	10,800	980
7 (2 gallons added)	14,700	2,180
8	14,300	1,760
9 (5 gallons added)	21,900	1,780
10	12,900	1,740
11	14,500	1,270

Figure 2 also shows when additions were made to the total fluid, and it can be seen that the treatment works well in providing a stable, consistent composition in the cooling fluid. Even though the analytical results looked promising, the fluid was deemed non-recyclable by the users because it "looked bad." Obviously, some method of either achieving a better appearance is required or the user needs to be made aware of the analytical results immediately in order to increase the likelihood of fluid reuse.

Figure 2. Microemulsion Fluid Composition as a Function of Time and Treatment (period dates indicate sample numbers)



COST/BENEFIT ANALYSIS

A representative from a local (Tulsa-based) waste disposal company was interviewed for information regarding metal fabrication shop wastewater disposal costs. To begin service with this company, a one-time start-up fee of \$250 is charged for analysis of a waste sample to characterize the waste and to determine optimum disposal methods. The charge for disposal is \$180 per 55-gallon drum. A pick-up charge of \$130 is incurred each time the disposal company removes the wastewaters, regardless of the number of drums. This encourages the small business to store large quantities of wastewaters on-site to avoid high hauling charges. The disposal company representative reported their average customer has wastewaters hauled 2 to 5 times per year.

The owner of a local, small (15 employees) metalworking shop was also interviewed regarding wastewater disposal costs. His company disposes of approximately six 55-gallon drums annually, and he reports the disposal costs are "very high." He is pursuing the possibility of purchasing an on-site recycling system at a potential cost of \$23,000. Clearly, a need exists for an inexpensive alternative for disposing of metalworking wastewaters by small businesses.

Based on the charges suggested above, a small operation disposing of three 55-gallon drums (1 drum 3 times) could incur the following costs as shown in Table 5.

TABLE 5. Cost Comparison of Disposal of Metalworking Wastewaters and Coalescence Treatment for Recycling

Disposal	
One-time analytical fee	\$250
Pickup charge (x3)	\$390
Disposal charge (x3)	\$540
Total	\$1,180
Anticipated costs for SRTA service	
Operating costs	~\$0.45/gallon/treatment
Amortized equipment cost (~ \$40,000; 7 years with 15 customers/month/year)	~\$32/customer/treatment
For treatment of a 25 gallon sample (10 per year)	
Operating cost (0.45 x 25 x 10)	\$112.50
Capital cost (32 x 10)	\$320.00
Profit @ 15% (0.15 x 432.50)	\$65.00
Total	\$497.50

This represents a savings of almost 58 percent to the user. The process could provide a real economic benefit to small users if it enables them to avoid disposing of their wastewaters. Even if SRTA's operating costs double (possibly from higher wages, longer treatment times, and the like), a significant savings over disposal could still be realized.

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Incentives

The user can pay a small monthly service charge to have his wastewaters treated, thus allowing him to recycle virtually all of the water. The incentives are lower cost, less potential for accidental discharge or mismanagement of hazardous wastes, and less problems associated with meeting state and federal regulations.

Barriers

The most immediate barrier is that although the fluid may be analytically clean enough to recycle, the operator, through lack of knowledge, may choose not to recycle due to visual quality. This can be overcome by providing adequate analytical results at the time of treatment to verify separation in order to build user confidence. This process is less effective on heavily contaminated fluids, since it does not provide complete separation.

A secondary barrier is a lack of experiential data to show to potential users. Continued development and field testing, which is being conducted, will overcome this problem.

REDUCTION OR ELIMINATION OF COOLING TOWER CHEMICALS

by

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ABSTRACT

Cooling towers used in air conditioning applications are a familiar site near many buildings. Since cooling towers are vast heat exchangers, the water in the tower becomes a perfect incubator to grow bacteria, algae, and fungi. Cooling towers are also plagued by corrosion, scale, and sediment accumulation. Water Equipment Technologies (WET) addressed these problems through an innovative process utilizing a zinc and copper alloy that is placed in the cooling tower sump. The reaction of the cooling tower water with the alloy raises the water's pH, and thus reduces corrosion, prevents algae from forming, and destroys bacteria. This concept reduces or eliminates the need for chemical algaecides, fungicides, corrosion inhibitors, and scale inhibitors currently used to maintain cooling towers.

INTRODUCTION

PROJECT DESCRIPTION

<u>Background</u>

In 1987, Don Heskett, an inventor, was experimenting with the removal of chlorine from water and inadvertently used a piece of brass to stir chlorinated water in a glass beaker. To his amazement, the chlorine disappeared. The brass consisted primarily of copper and zinc with smaller quantities of lead, iron, and cadmium. Since lead and cadmium are undesirable metals in water supplies, a special alloy of zinc and copper -- named KDF -- was formulated to achieve the same results.

Further tests have shown that a mixture of highly pure, molten zinc and copper yields an alloy with millions of bimetallic couples. The zinc acts as the anode and copper acts as the cathode. Exposing sufficient surface area of the alloy to water creates an electrical potential (Eh). The Eh for zinc is -0.76 milli-volts, and the Eh for copper is +0.34 milli-volts. Zinc becomes the electron donator and the resulting redox reaction -- corrosion -- causes electro-chemical reactions that provide a "natural" treatment process to control the formation of hardness scale, bio-film development, chlorine removal, and ionic heavy metal reductions.

KDF comes in several forms (e.g., granules, filament or wool, wire or brush, and powder) with varying levels of pure copper and zinc, depending on the application. The granules are used in backwashing-type filters much the same way sand is used to filter water, except that KDF also removes chlorine, bacteria, and heavy metals. The wool is used on recirculation loops, lending itself to applications such as cooling

towers. The brush form is for small portable devices where the removal of chlorine after disinfection is desired. The powder provides faster reaction times in treating wastewater (and is currently under development).

The KDF Process

The KDF process is as follows:

- 1. Chlorine is removed through its conversion to zinc chloride (Cu/Zn + Cl_2 = Zn^{++} + 2Cl'). Zinc loses 2 electrons through oxidation, and chlorine gains 2 electrons through reduction.
- 2. Heavy metals are removed by plating-out the metals onto the cathode (copper) sites $(Cu/Zn + Pb^{++}(NO_3)_2) = Zn/Cu/Pb + Zn^{++}((NO_3)_2)$. Zinc loses 2 electrons through oxidation, lead gains 2 electrons through reduction and plates-out on the copper sites. Excess zinc ions have a tendency to redeposit back onto the media.
- 3. Calcium carbonate precipitation and scaling are controlled by an undetermined mechanism that reduces the calcium carbonate's ability to link together. The electrochemical reactions interfere with the crystalline structure of limescale, and a powdery, rather than vitreous, scale formation is seen when the water dries in the splash areas of the cooling tower. No scale is noted on the heat transfer surfaces, and no chemical formula has been developed to explain this phenomenon.
- 4. Bio-film accumulation and bacterial growth are controlled by the formation of hydroxyls. Some of the water reacts with the zinc that liberates a small portion of the hydrogen ion from water molecules, causing hydroxyls to form (Zn + 2H₂O = Zn(OH)₂ + H₂). WET believes that these "OH" radicals, along with redox shock, interfere with the normal cellular activity of bacteria and algae, thus reducing bio-film formation. Redox shock is the change in electrical (Eh) potential. As a rule, different types of bacteria and algae can only grow within a particular range of redox potential. The passage of water through KDF causes a rapid and reversible reduction in Eh value of about 500 millivolts. This redox shift results in a disruption of electron transport and possibly causes a cascade of subsequent damage to the cell walls of single-cell organisms.
- 5. Corrosion inhibition is achieved by the less-nobel metal, zinc, "sacrificing itself" to protect the other metals. In the case of KDF, the zinc ions in solution are available to react chemically or to sacrifice themselves to protect other metals in contact with the water.

Outline of Process/Product

In the United States, over 700,000 cooling towers use water to cool buildings, process equipment, or products. Water is pumped through a heat exchanger, transferring the heat into the water. This water enters the top of the cooling tower, passing into the fill areas and through forced air that evaporates some of the water. The evaporating water cools the remaining water (by approximately 8 to 15 degrees fahrenheit), which then re-enters the heat exchanger.

If scale or algae forms in a cooling tower, efficiency is reduced, and dangerous bacteria (e.g., legionella) may develop. Also, the dissolved salts that remain when pure water evaporates can form scale that interferes with the transfer of heat. High salt contents become corrosive to the metal pipes, condensers, and structure of the cooling tower, which can cost millions of dollars in failed equipment and repairs each year.

A multitude of chemicals (e.g., chromates, phosphates, silicates, pesticides, herbicides, biocides) have been developed to treat cooling tower maintenance problems. During normal operation of the towers, these chemicals can enter water ecosystems and water treatment plants. Many chemicals have already been banned due to their toxicity, but some chemicals must still be used to address these problems.

WET tested the KDF wool media as a replacement for chemicals to manage algae, bacteria, scale, and corrosion in cooling towers. The test site was a comfort cooling tower in Fort Lauderdale, Florida. Part of the water flowing through the tower system passed through floating modules contained KDF wool that were placed in the basin. The redox reaction occurred on a continuous basis during the daily operation of the tower. The flow through the KDF was approximately 6 percent of the total tower flow per minute.

<u>Unique Product Features/Advantages</u>

KDF wool provides a natural control mechanism that raises the pH of the water, which then reduces redox activity in a self-regulating mode.

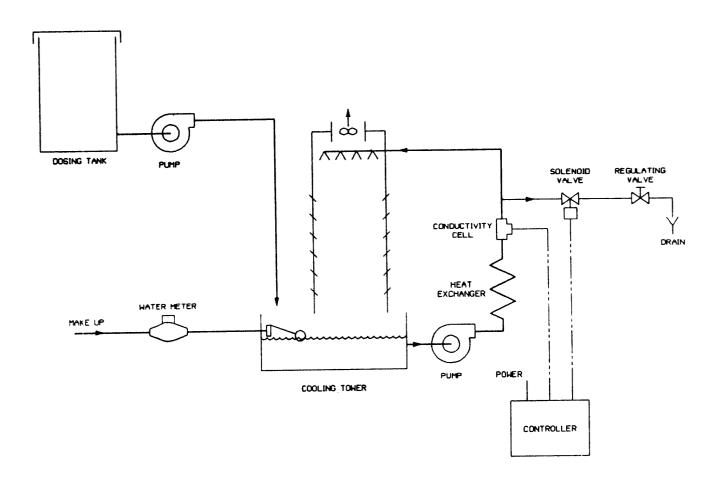
KDF is a unique, but common alloy, and once spent, the material may be recycled through local scrap dealers.

Process Schematic

Cooling towers can vary in size from 8 to over 10,000 tons in capacity. Water flow rates can range from 35,000 to 43,000,000 gallons per day.

A typical multi-chemical treatment (MCT) cooling tower is shown in Figure 1. Water enters the cooling tower on demand through the make-up water meter. A circulating pump pushes the tower water through a heat exchanger and into the cooling tower section where the water releases the heat acquired in the heat exchanger. Chemical dosing pumps feed various chemicals into the tower basin, while a controller measures the salt content of the water via a conductivity cell. When the salt content reaches a certain level due to evaporation, the bleed manifold valve automatically opens, and the water and chemicals are discharged to drain.

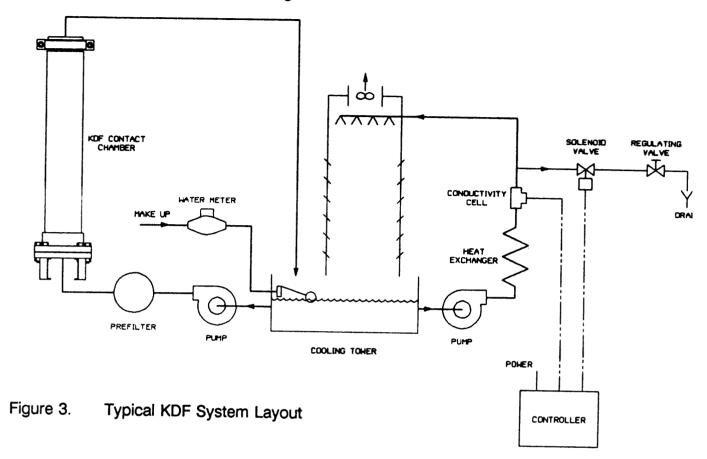
Figure 1. Chemically Treated Cooling Tower

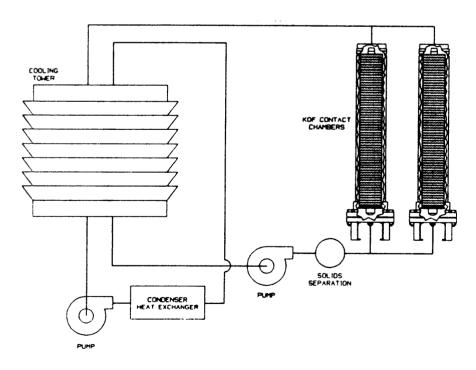


A KDF wool system (KWS) cooling tower is represented in Figures 2 and 3. Make-up water enters the cooling tower via a float valve. The cooling tower circulation pump sends water through the heat exchanger and back to the tower for cooling. A Total Dissolved Solids (TDS) controller measures salt content through a conductivity cell, and a solenoid valve opens to release non-chemically treated water to drain. A separate low-volume pump recirculates cooling tower water through a prefilter to remove solids, then through a KDF wool contact chamber, and into the basin of the cooling tower. Chemical dosing pumps are not used.

Initially, KDF wool was placed in floating modules that had slots to allow water to flow across the wool. The modules were placed in the cooling tower basin. Water either fell into them directly from the fill area, or water was pumped into the modules so that water was recirculated on a 24-hour basis. When sediments began filling and sinking the modules, a second generation containment vessel was developed. The new "contact chamber" was located next to and outside the cooling tower basin. Water was filtered and then pumped into the chamber. The water reacted with the KDF wool media and flowed back into the cooling tower basin.

Figure 2. KDF Treatment Flow Diagram





APPLICATION

Process/Product Replaced

KDF wool replaces traditional cooling tower chemicals. According to the March 1993 issue of *Industrial Water Treatment* magazine, "Over \$750 million dollars worth of cooling tower chemicals are used to treat approximately 500,000 "comfort" cooling towers in the U.S. These towers are used for cooling hotels, resorts, and offices. These figures do not include the tens of thousands of industrial and utility cooling towers that use billions of gallons of water each year." Some of the chemicals that could be replaced include:

- Organophosphorus compounds
- Polyacrylate compounds
- Polymethacrylate polymers
- Chromates
- Polyphosphates
- Nitriles
- Silicates
- Heterocyclic compounds (mercaptobenzothiazole, benzothiazole)
- Chlorine
- Organosulphur compounds
- Bisthiocyanates
- Organobromines

Wastes Prevented

Treatment chemicals would be reduced or eliminated with the use of KDF wool.

The number of labor hours needed to monitor and maintain cooling towers could be halved by using the self-regulating KDF wool media. As KDF raises water pH, it slows its own activity. As the lower pH make-up water enters the cooling tower basin, increased activity of the KDF raises the pH and once again stabilizes the KDF process.

Water use is also reduced. Chemically treated towers may drain constantly to prevent scale and algae build-up. Since KDF wool is self-regulating, discharge rates may be significantly reduced. Also, tests have shown that KDF towers can operate at higher salt concentrations than chemically treated towers, which results is less water usage.

Cross Segment Uses

KDF wool could be used in a variety of water treatment applications:

Cisterns or water storage tanks -- KDF wool can be used in drinking water systems to reduce bacteria. For example, many countries use roof-top tanks to store drinking water for a building's occupants. These tanks become havens for biological activity. EPA permits KDF to claim bacteria-static properties and has classified KDF as a pesticidal device, not a pesticide.

- Medical -- Hospitals use water for a variety of applications, such as water immersion therapy for burn patients. A patient's potential for bacterial infection is enhanced when the skin's protective barrier is removed. KDF wool-treated water could eliminate harmful bacteria from the water prior to patient treatment.
- Hot water systems -- Many hot water systems harbor bacteria, such as legionella, which thrive in 140 degree fahrenheit water. Laboratory tests found that legionella bacteria died when exposed to water that has been passed through the KDF.
- Heavy metal reductions -- Heavy metals are often difficult to remove during the
 wastewater treatment process. KDF can remove part per billion levels of certain
 heavy metals. Tests by the New Jersey Department of Health showed that KDF
 is the best technology for removing mercury from water.
- Chlorine removal -- Many wastewater treatment systems contain chlorine that can be expensive to remove. Tests on a secondary wastewater stream in St. Charles, Illinois, found that 57 pounds of KDF processed over 1 million gallons of effluent while removing all chlorine at a flow rate of 5 gallons per minute.

PROCEDURE

DEMONSTRATION

Two existing circular, fiberglass Protec comfort cooling towers, located at a mid-sized hotel in Fort Lauderdale, Florida, were selected for the project.

The cooling tower on the ground floor level of the hotel is rated at 700 tons, and the roof tower is rated at 225 tons. Both towers were being chemically treated by a service contractor. For the trial, the ground-level tower continued to be chemically treated for comparison with the KDF wool-treated tower on the roof. WET elected to use the roof tower for the KDF treatment since it offered better security against vandalism.

The test tower was pressure washed to remove visible scale and algae. The basin was vacuumed, drained, and refilled. The pH of the water was reduced to 7.0, and eight, 10-gallon-per-minute floating modules containing KDF wool were installed in the basin so they would capture the natural flow of water cascading through the fill area.

Two months prior to the start of testing, base-line corrosion coupons -- small strips of different types of metals -- were installed in both cooling towers. These monitor corrosion rates for mild steel, galvanized steel, admiralty brass, stainless steel, and rolled copper. The coupons were immersed in water for a period of time and then sent to a laboratory for corrosion analysis.

Special biofilm coupons, developed by Dr. John Wireman of Biological Solutions, were also installed to monitor the growth of biofilms. The coupons were made from a material that allowed biofilms to attach to the coupon surface. Measurements between the two towers could be compared.

For twelve months, WET technicians conducted site visits every two weeks to monitor water chemistry -- hardness, TDS, copper, zinc, iron, alkalinity, temperature, and pH -- and note conditions of the towers. The corrosion coupons were replaced every two months; used coupons were sent to an independent laboratory for analysis. Biofilm coupons were collected on a monthly basis and sent to Dr. Wireman for analysis.

During the last eight months of the project, other KDF wool cooling tower sites were also being monitored. At these sites, WET charged the owners the same fee as they were paying for chemical treatment (as EPA funding did not cover these sites) and guaranteed equal or better results. WET was concerned that, due to a vandalism incident of the test roof-top tower, and damage to the tower inflicted by Hurricane Andrew, they needed additional data to better understand the results obtained from the Grant Program test site.

Evaluation Parameters

The KDF and chemically treated cooling towers were compared for corrosion rates, bacterial growth, and scaling tendencies.

Assessment Summary

KDF wool controls scale and biofilm, and to a lesser degree, corrosion. A vandalism incident and damage inflicted by Hurricane Andrew did not permit a 12-month uninterrupted test cycle at the EPA-funded test site. However, from information collected at the 12 other test sites, WET concluded that the average performance life of KDF wool on South Florida water is 6 months.

Cost/Payback of Demonstration

The final project cost was \$42,297, with \$24,830 contributed by EPA through the Pollution Prevention By and For Small Business grant program; WET contributed the balance of \$17,417.

The cost to chemically treat the test tower would have been approximately \$1,800. The cost to use KDF on a normal basis would be comparable.

Although WET had to replace the KDF media 3 times during the test project due to various upsets, this led to the development of a second generation containment vessel to hold the KDF media. In addition, WET learned to filter the water to remove large particles of scale and dirt before passing the water through the media.

WET can offer the KDF technology at a similar price to chemical treatment, but with less environmental impact, less water usage, and reduced labor.

RESULTS AND DISCUSSION

The purpose of the pilot study was to determine the efficacy of KDF wool process media in lieu of traditional multi-chemical methods in the treatment of

problems common to water-based heat exchange systems. During the study, data were collected in the following areas:

- Corrosion rates
- Bacteria
- Langelier Scaling Index values
- Cycles of Concentration
- General water chemistry

The pilot also allowed an evaluation of media application to similar systems.

Several problems developed during the course of the pilot study. In late May 1992, the site was subjected to tampering by unknown parties. Concurrently, a recurring mechanical problem with the tower's rotating distribution arm developed. In late August 1992, Hurricane Andrew passed through south Florida, damaging the site and causing system upsets. In the third quarter of the pilot (late 1992), the distribution arm malfunction was causing a serious problem, and termination of the study was considered if repairs were not made by the tower owner. The tower distribution arms were repaired in mid-January 1993.

A cooling tower (not in the pilot study) was retrofitted in August 1992 with a new method of media application that utilized a newly developed contact chamber and a side-stream approach; this yielded better results than using KDF in the floating modules. Due to site specific restrictions, WET could not replace the modules with a contact chamber at the pilot tower.

Of the general parameters studied during the pilot period, the KDF data indicate a favorable comparison with the multi-chemical treatment methods used on the control tower. Bacterial and corrosion rate data between the two towers indicate comparable results except as noted in the following discussions. Water chemistry analysis conducted during the pilot period was similar, as seen in the Langelier Scaling Index (LSI) values and Cycles of Concentration data.

Test data from only one site cannot be considered conclusive for all cases, and as water chemistries change, results may vary. This initial study indicates comparable results between the KWS and MCT methods and highlights the KDF wool technology as a viable alternative to chemical methods that adversely impact the environment. Further areas of research and development are anticipated with specific interest in a more in-depth study of heat transfer efficiencies, biofilm formation, corrosion rates in higher cycles of concentration applications, and a closed-loop application that would allow a zero discharge from the cooling tower.

PRODUCT QUALITY VARIANCE

The floating modules performed inconsistently and did not promote an even water flow through the KDF media. WET then introduced the external contact chamber that used well screens wrapped with KDF wool. Water was pumped through the contact chamber and KDF while a flow restrictor maintained optimum flow conditions.

CONDITIONS THAT IMPACT PERFORMANCE

pН

Redox reactions were optimized when water pH was below 8.5. When the KDF wool reacts with the water, pH was raised and the KDF process slowed. The addition of lower pH feed or make-up water reactivated the KDF reaction.

<u>Air</u>

Once the KDF wool is wet, exposure to air will shorten its performance life. When the test tower was vandalized and drained, and when the tower was damaged by Hurricane Andrew, the wet KDF wool was exposed to air and dried. The wool never regained optimum performance and had to be replaced.

Sediment

A build-up of sediment in the KDF media only traps more sediment, and the KDF becomes a filter rather than a redox reactor.

Hydraulics

A steady, even flow of water over the entire KDF media surface is necessary to optimize performance. An ideal flow rate is 10 gallons per minute for 5 pounds of KDF wool.

TABULATION OF DATA

LSI Values and Cycles

This data illustrate the relationship of the LSI and the Cycles of Concentration. The LSI is commonly used as a point of reference relating to the aggressive (or scaling) nature of the system water. A range of \pm 1.0 indicates a relatively neutral water, not necessarily aggressive or scaling. Cycles of Concentration is a relative term referring to the mineral concentration(s) of the system water versus the source water. The LSI and Cycles of Concentration are important, as they provide a picture of the system that cannot be directly observed or quantified through other means. With Cycles of Concentration, as "mineral levels left behind by the evaporation of pure water" increase, the aggressive nature of the system water may be minimized, and scaling tendencies become pronounced. However, high levels of chlorides will add to the aggressive nature of the system water and contribute to corrosion rates. The Cycles of Concentration also indicate the number of times the system water is reused before it is discharged to drain. Thus, the higher the cycles, the more efficient the use of water in the tower.

During the pilot period, WET was not aware of any scaling in the heat exchange areas. The average Cycles of Concentration for the KWS tower was 4.8353, and the average LSI value was 1.1106. The average cycles for the MCT tower was 4.62, with an average LSI value of 1.1677. The performance of the KWS tower was comparable to the performance of the MCT tower. A comparison of LSI values and cycles in the KWS tower and the MCT (control) tower are shown in Figures 4 and 5 and in Table 1.

Figure 4. Pilot Tower - KWS: LSI Values and Cycles

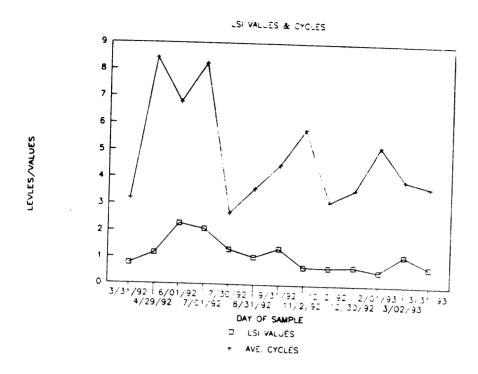


Figure 5. Control Tower - MCT: LSI Values and Cycles

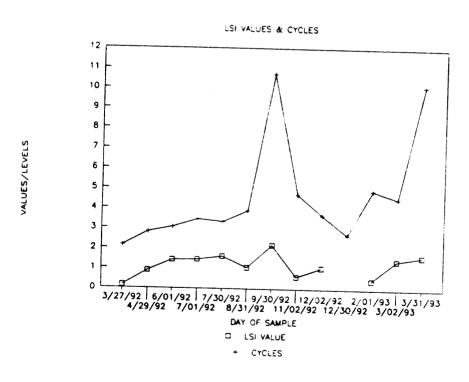


TABLE 1. KWS (Pilot) and MCT (Control) Towers: LSI Values amd Cycles

	KWS TOWER				MCT.T	OWER	
Sample Date	ဇ	LSI Value	Total Averag e Cycles	Sample Date	ဇ	LSI Value	Total Averag e Cycles
3/31/92	24.4	0.7699	3.20	3/27/92	27.0	0.1535	2.16
4/29/92	25.8	1.1677	8.44	4/29/92	25.8	0.8846	2.82
6/1/92	27.9	2.2842	6.79	6/1/92	27.1	1.4225	3.07
7/1/92	28.0	2.0910	8.24	7/1/92	27.8	1.4458	3.45
7/30/92	30.5	1.3217	2.69	7/30/92	28.7	1.6165	3.33
8/31/92	28.1	1.0326	3.61	8/31/92	27.9	1.0553	3.88
9/31/92	26.3	1.3677	4.49	9/30/92	26.7	2.1977	10.72
11/2/92	27.6	0.6853	5.82	11/2/92	27.1	0.6141	4.72
12/2/92	28.5	0.6639	3.13	12/2/92	27.1	1.0580	3.69
12/30/92	25.4	0.7007	3.58	12/30/9 2	26.3		2.72
2/1/93	19.8	0.5270	5.21	2/1/93	23.6	0.4847	4.94
3/2/93	24.8	1.1319	3.95	3/2/93	24.1	1.4463	4.53
3/31/93	27.2	0.6945	3.71	3/31/93	27.9	1.6528	10.14

Bacterial Data

The relationship between the total bacterial plate counts in the KWS tower and the MCT tower is shown in Figure 6 and in Table 2. Initially, the KWS tower yielded much lower total plate counts than the MCT tower. With a system upset due to tampering in May 1992, bacterial counts were still lower than those seen in the MCT tower. However, counts spiked during August to September 1992, and KWS bacteria counts exceeded MCT counts.

The first indications of system water distribution problems in the test tower surfaced in mid-July 1992. Since the distribution arms were not rotating, a full flow of system water through the KDF media was not possible. Bacterial counts began to rise. In late August 1992, Hurricane Andrew caused a massive system failure, and the tower was pumped dry on several occasions, exposing the wool and wetted surfaces to ambient air. The tower was also inoperable for several days with no flows through the wool media. When stabilized and operational, WET noticed a significant drop in total bacterial counts from the September 30, 1992 levels. However, from that point,

The annual average of the total bacterial counts were within 9 percent of values from the test and control towers. A significant deviation in bacterial counts in the KWS tower was noted during the August/September 1992 period. If this count of 2.8E6 were at a level of 1.5E6, consistent with the MCT tower, the annual average figure for the KWS tower would have been approximately 666,182 colony forming units (CFU) per month or 9 percent less than the annual average for the MCT tower. From the data presented, the total bacterial counts reported in the KWS tower were well within the ranges of those seen in the MCT tower.

Figure 6. KWS and MCT Bacterial Plate Counts

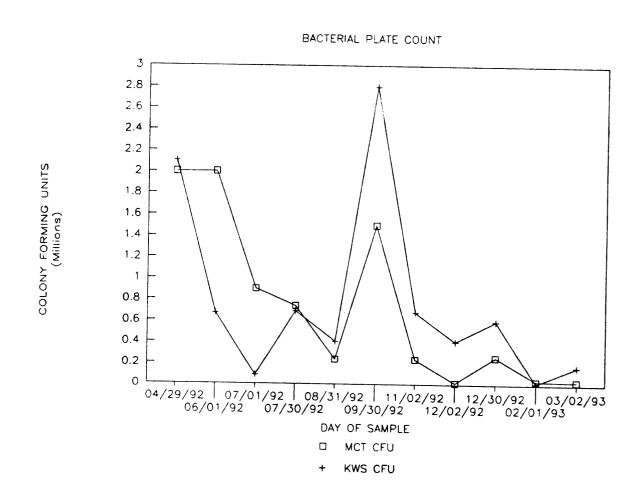


TABLE 2. KWS and MCT Bacterial Plate Counts

DATE	MCT TOTAL BACTERIA	KWS TOTAL BACTERIA
4/29/92	2,000,000	2,100,000
6/1/92	2,000,000	670,000
7/1/92	900,000	85,000
7/30/92	740,000	690,000
8/31/92	240,000	410,000
9/30/92	1,500,000	2,800,000
11/2/92	240,000	680,000
12/2/92	16,000	410,000
12/30/92	260,000	600,000
2/1/93	36,000	13,000
3/2/93	33,000	170,000

Corrosion Rates - Alloy

Corrosion rate data compare the corrosion rates in the KWS and the MCT towers on alloy metals. These metals, due to their composition, are less susceptible to corrosion in most environments and are representative not only of the metals in this system, but of HVAC systems in general. Upon initial observation, the corrosion rates in the MCT tower are subject to radical fluctuations. This may be due, in part, to the adjustment of chemical feeds for pH and inhibitor control in response to the changing nature of the source water chemistry. The self-regulating nature of the KDF, in response to pH changes in the system/source water, allows for a more consistent level of corrosion inhibition. KWS tower base-line corrosion figures gathered prior to the initiation of the pilot indicate a rate of 0.31 mils per year for rolled copper using CDA 122 coupons. The average annual corrosion rate for the same coupon during the test with KDF wool was 0.431 mils per year; the annual MCT tower corrosion rate for copper was 0.530 mils.

Base-line data were not collected for admiralty brass or stainless steel coupons. During the test period in the MCT tower, the average annual corrosion rate for admiralty brass was 0.677 mils, and 0.241 mils for 316 stainless steel. In the KWS tower, corrosion rates were 0.798 mils for admiralty brass and 0.249 mils for stainless steel. The KWS tower performed favorable as compared to the MCT tower. Corrosion rates in the KWS and MCT towers are shown in Figures 7 and 8 and in Table 3.

Figure 7. Pilot Tower - KWS: Corrosion Rates

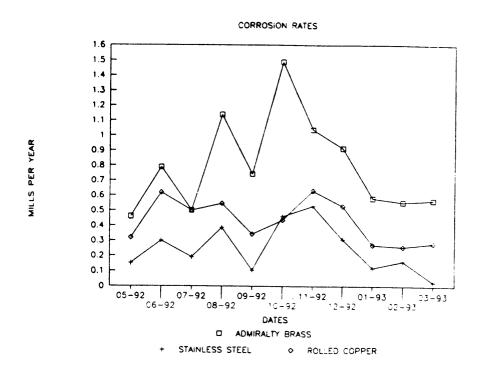


Figure 8. Control Tower - MCT: Corrosion Rates

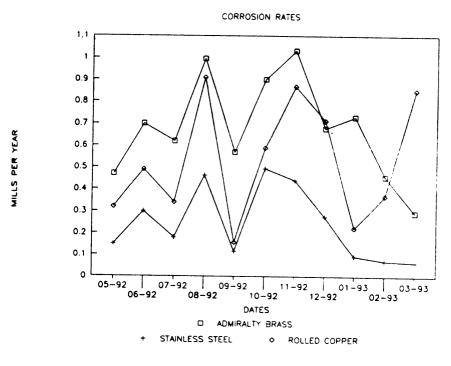


TABLE 3. KWS and MCT Towers: Alloy Corrosion Rates

KW	KWS Alloy Corrosion Rates			MCT Allo	y Corrosic	on Rates
Date	Adm. Brass (mils)	Stain. Steel (mils)	Rolled Copper (mils)	Adm. Brass (mils)	Stain. Steel (mils)	Rolled Copper (mils)
5/92	0.46000	0.15000	0.32000	0.47000	0.15000	0.32000
6/92	0.79000	0.30000	0.62000	0.70000	0.30000	0.49000
7/92	0.50000	0.19000	0.50000	0.62000	0.18000	0.34000
8/92	1.13900	0.38600	0.54570	0.99530	0.46280	0.90870
9/92	0.74260	0.10380	0.34260	0.57030	0.11610	0.15780
10/92	1.48840	0.45910	0.43670	0.90000	0.49450	0.58940
11/92	1.03750	0.52700	0.63060	1.03260	0.43980	0.86810
12/92	0.91330	0.30930	0.52830	0.67930	0.27570	0.71350
1/93	0.58090	0.11930	0.27140	0.73080	0.09360	0.22350
2/93	0.55530	0.16320	0.25940	0.45750	0.07170	0.36960
3/93	0.56810	0.02850	0.28260	0.29370	0.06540	0.85030

Corrosion rate tests (measured in mils per year) for the KDF tower indicate similar results for admiralty brass, stainless steel, and rolled copper, but the KDF tower showed less variation in the acceleration of corrosion rates. WET feels this is due to the constant adjusting of chemical dosing to match the ever changing feed water supply (in chemically treated towers).

Corrosion Rates - Steel

The chemically treated towers in the pilot study have lower corrosion rates for mild steel and galvanized steel. The data for steel corrosion rates indicate the relationships between the KWS and MCT towers on untreated, C1010 mild steel coupons. Black iron coupons may be considered more representative of system metallurgy; however, the difficulties of correctly determining corrosion rates on this type of coupon outweigh the prospective benefits of this data. The HD galvanized coupons are representative of system metallurgy.

Base-line corrosion rate data were collected from the KWS tower under chemical treatment conditions prior to the initiation of the pilot study on both mild steel (7.05 mils) and HD galvanized (4.95 mils) coupons. During the pilot test, the average annual corrosion rates in the MCT tower were 9.225 mils on the mild steel coupons and 4.250 mills on the HD galvanized coupons. When treated with KDF wool, annual corrosion rates in the KWS tower were 16.418 mils for mild steel coupons and 5.889 mils for HD galvanized coupons.

Concentration with a similar source water chemistry. The results of the test indicated an HD galvanized corrosion rate of 3.820 mils per year. This is not conclusive, yet it may be presumed that at higher Cycles of Concentration, the corrosion protection of milder metals would be better. This is contrary to what one would normally expect to see under such conditions.

A comparison of corrosion rates in the KWS and MCT towers may be seen in Figures 9 and 10 and in Table 4.

Figure 9. Pilot Tower - KWS: Steel Corrosion Rates

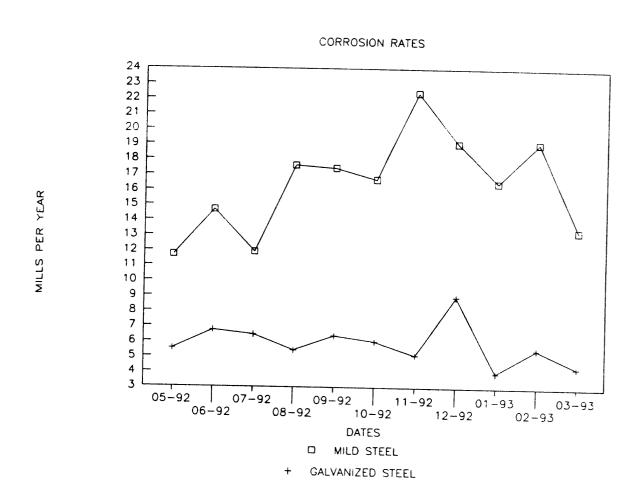


Figure 10. Control Tower - MCT: Steel Corrosion Rates

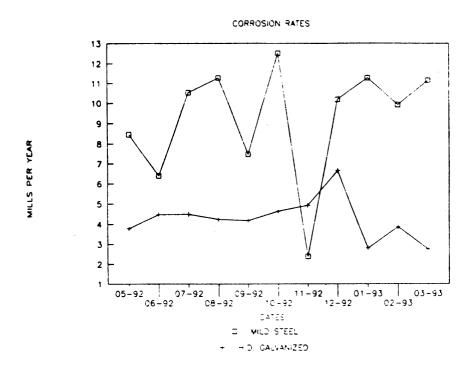


TABLE 4. KWS and MCT Towers: Steel Corrosion Rates

KW	KWS Steel Corrosion Rates			MCT Steel Corrosion Rates		
Date	C1010 Steel (mils)	HD Galvanized (mils)	C1010 Steel (mils)	HD Galvanized (mils)		
5/92	11.71000	5.54000	8.45000	3.79000		
6/92	14.70000	6.75000	6.39000	4.47000		
7/92	11.92000	6.48000	10.52000	4.49000		
8/92	17.61000	5.45500	11.26400	4.23800		
9/92	17.43180	6.40770	7.48710	4.17340		
10/92	16.71370	6.04510	12.51040	4.63290		
11/92	22.40580	5.14750	2.37490	4.91410		
12/92	19.09190	9.00530	10.17310	6.64450		
1/93	16.52610	4.00400	11.27130	2.79700		
2/93	19.12480	5.55950	9.91520	3.85400		
3/93	13.36260	4.38450	11.14290	2.7415		

Biofilms

During the first two months of the test, the KDF wool tower showed a remarkable ability to prevent the growth of biofilms as compared to the chemically treated tower.

At the end of the second month, vandals drained the cooling tower, exposing the KDF wool media to the air. From that point, biofilm tests did not show reduction levels as they had previously. After 4.5 months, the KDF wool was replaced, as results from the vandalized tower were still substandard as compared to the other KDF towers. In August 1992, Hurricane Andrew damaged the tower, causing the water to drain and exposing the KDF wool to the air. Again, the KDF wool was replaced.

Recurring mechanical problems with the water distribution arms on top of the tower was causing uneven flows through the floating KDF modules. The KDF wool was replaced in October 1992, and by December 1992, biofilm tests still had not improved as expected. In January 1993, building site management repaired the malfunctioning water distribution arms. Fungi counts in the KDF tower then dropped to half the level of the chemically treated tower, although algae counts were higher.

Given the results of treatment generated through the use of the contact chamber design on other sites of this nature, WET made a decision to discontinue the use of the floating modules in this application.

Scale Control

Once the KDF wool was installed in the test tower, the amount of scale that broke free from the interior of the fill area and distribution system was high. The debris sank several floating modules. Water hardness tests indicated that scale did not seem to form, even when the level of water hardness reached normal saturation or precipitation levels.

COST/BENEFIT ANALYSIS

Costs to treat a cooling tower with KDF and with chemicals are virtually the same, and the owners/operators of cooling towers would generally accept this new non-chemical approach at the same value level they pay for chemical treatment services. For the test tower, annual treatment costs totaled \$1,800 for each type of treatment.

Certain benefits for the cooling tower owner may be derived from using KDF media:

- No chemicals stored on-site
- No chemicals discharged into the environment
- Better scale control, which reduces energy use
- Enhanced pro-environmental image
- Reduced liability risks for chemical management

Benefits for the cooling tower service company include:

- No chemicals to mix or transport
- No chemical metering pumps to fail or malfunction
- More consistent treatment for customers
- Reduced labor costs by a contracting company
- Ability to recycle the spent KDF material
- Comparable profit structure
- Ability to increase territory and client base since fewer labor-hours are required to monitor and control KDF technology
- Reduced occupational hazards for employees (as compared to chemical treatment methods)

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Incentives

The KDF technology is already patented. However, in some countries that do not honor USA patents or even their own native patents, the technology could be copied.

Advanced Water Management Group, a division of WET, has been established to expand the technology in the Florida cooling tower market. Future activities include introducing the KDF technology to comfort tower service companies who currently use chemical treatment systems. Advanced Water Management Group also hopes to gain the interest of tower manufacturers and chemical producers who serve the cooling tower industry.

Barriers

Competitive challenges of the marketplace and cash flow are normal barriers to any technology. WET needs additional funding to carry the KDF technology into the larger industrial and utility cooling tower market. WET must also educate cooling tower operators and service companies of the merits of this new, non-traditional approach to maintaining cooling towers.

PRE-CHARGED VACUUM LIQUID EXTRACTOR / CONTAINERIZATION DEVICE

by

Lowell Goodman Technical Support Services SOUTH THOMASTON, ME 04858

ABSTRACT

Technical Support Services has developed and tested an innovative device that provides simple, spill-free extraction of used oil from equipment during routine maintenance activities such as engine oil changes. The objective of this project is to design, test, and evaluate this device for use in other applications. Successful development of this device will provide a mechanism to remove and contain materials in otherwise diffucult extraction situations, thereby promoting recovery and recycling of material which is frequently disposed.

INTRODUCTION

PROJECT DESCRIPTION

The Slurper™ is an innovative, self-contained, pre-charged vacuum device which provides simple, spill-free extraction of fluids - primarily used oil from marine engines and industrial equipment during routine oil changes. Liquid is "vacuumed" into a sealed, steel tank for spill-proof containerization until the material is reclaimed. The objective of the project is to design, test, and evaluate modifications of this device for other applications such as recovery of chlorofluorocarbons (CFCs), extraction of automotive fluids from vehicles at an accident scene or salvage yard, and extraction and containment of bodily fluids by embalmers. Removing and containing hazardous liquids in difficult extraction situations can promote recovery and recycling of materials which may ordinarily be disposed. The project included design work, operational and flow rate testing, and analytical work and engineering studies.

Two single compartment models were designed. A 4.75 gallon model serves those uses which require an extraction capacity of less than 18 quarts (such as automotive engines). A 9 gallon design accommodates larger capacities, such as diesel engines which typically have a 5 gallon oil reservoir. The latter unit is targeted for commercial applications. Both units are configured with standard 3/4 inch drain valves for easy draining and cleaning.

A two-compartment unit was also designed to remove waste oil by vacuum and replace fresh oil by pressure. (This concept evolved prior to receipt of the Grant and was submitted with the original Slurper™ patent application.) This unit has two sides that perform the following functions:

- "Take in" fresh oil by vacuum
- Remove waste oil by vacuum
- Replace fresh oil by pressure

In the two compartment model, both halves are precharged with vacuum. A suction tube is connected to what is normally the "pressure side" of the vessel, allowing fresh oil to be "taken in" by vacuum. Next, air pressure is applied to the "pressure side" on top of the fresh oil. The unit is now ready to draw in waste oil via the vacuum side and replace fresh oil via the pressure side.

Unique Product Features

The vacuum process can simultaneously remove and containerize a material, thereby immediately preparing it for recovery. Key advantages include:

- Application of a portable self-containerized vacuum
- Simultaneous containment of liquids or gases
- Safe in explosive environments (no electricity)
- Application of a near absolute vacuum
- Chargeable by shop air pressure
- Extraction to metal hand operated connectors (vacuum tight)
- 97 percent fill capacity
- Light weight (no heavy pumps or batteries)
- Doesn't get "tired"

The last point is especially evident with highly viscous fluids. The Slurper™ keeps drawing on the material with the same force until fill capacity is reached without regard to time required to extract the material. There is no additional vacuum loss with a thicker liquid; vacuum loss is dependent only on the amount of liquid actually taken in. Figure 1 illustrates a one-compartment Slurper™. Figure 2 shows the two-compartment model.

The figure below outlines the Sturper** part by part. Numbers have been assigned to

Figure 1. One-compartment Slurper™

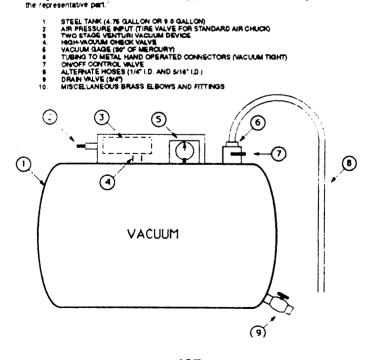
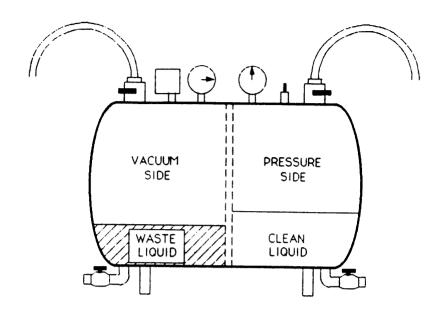


Figure 2. Two-compartment Slurper™



APPLICATION

Process/Products Replaced

For marine oil changes, the SlurperTM replaces conventional manual and 12 volt electric pumps mounted on a plastic bucket with an opening in the lid. These existing commercial pumps are low pressure fluid pumping devices and by design are inherently messy because the oil actually goes "through" the pump itself. These pumps function by pushing air forward which creates a "siphon" in the suction hose to initiate the pumping process. Once the oil is pumped into the bucket, the 12 volt motor must be reversed to pump the oil from the bucket; the oil can also be poured through the opening in the lid. Most pumps require close proximity to a battery or electric source for power, thereby limiting their portability. The electrically operated units are potentially hazardous in the bilges of boats with gasoline engines.

Wastes Prevented

No-spill vacuum containerization of used oil can enhance its recyclability, as containment, storage, and transport is simplified. The containment process eliminates the introduction of oil into the environment, as the oil is transferred directly from the engine into the Slurper™. Storage and transportation have no risks of spills.

With any liquid, if containment and storage are time consuming, awkward, or expensive, the individual or business may default to a more simple, but improper disposal method. For example, it is easier to pour old oil from a bucket into the sewer, rather than risk spilling oil in the trunk of one's car during transport to the proper recycling site.

Cross Segment Uses

Industries finding useful Slurper™ applications include: plumbing and heating contractors, trucking and fleet companies, rental vehicle fleets, automotive repair stations, and machinery in virtually any industry.

Innovative applications include:

- Embalming Embalmers do not always receive notification of an HIV positive cadaver, and concern over safe management of bodily fluids is prevalent in the funeral industry. Vacuum technology permits the evacuation and containment of bodily fluids during the embalming process.
- Holding Tank Cleaner Boats are not permitted to discharge the contents of the holding tank overboard. The Slurper™, modified with a larger diameter hose, can easily evacuate the tank.
- Welding Solder Extractor Excess solder can easily be removed from printed circuit boards and containerized for subsequent recycling.
- Oil Furnace Priming When the Slurper™ nozzle is attached to the bleed point on an oil furnace, priming may be quickly accomplished (especially during cold, winter months) when compared to traditional gravity bleed priming.

PROCEDURE

DEMONSTRATION

Test Conditions

Given the broad spectrum of materials which could be handled by the vacuum technique, tests and evaluations were conducted with:

- Liquid Containment
 - Water (control liquid)
 - Gasoline
 - Kerosene
 - Linseed Oil
 - Motor Oil (SAE 30)
 - 85/140 Gear Lube
- Gaseous Containment
 - Air
 - CFCs (Freon)

The liquid and gaseous tests encompassed percentage fill capacities and flow rates (suction characteristics). They were also designed to demonstrate operational boundary conditions for liquid containment within the containment sizes available. These basic designs were operationally tested to provide baseline information on performance and potential.

Flow rate tests were performed on a representative sample of materials. These tests were structured and controlled to provide representative data and to describe relationships among the operational parameters which significantly affect each of the flow rates.

Given the complexities of fluid dynamics, a thorough test series of all fluid dynamic factors was considered outside of the scope of the project.

The tests and test results for liquid flow are not quantitative to the Bureau of Standard's laboratory conditions, particularly in temperature control.

Boundary Conditions

Several tests were designed to present low end boundary conditions, where possible. For example, 85/140 gear lube was tested at 0° F with a 7 foot long, 1/4 inch internal diameter (ID) tube.

In designing boundary condition tests for technique, "everyday products" were tested to provide quick representation of low end response for highly viscous materials.

Repeatability was consistent throughout the liquid tests. Test results variations were mostly attributed to accuracy of gauge readings, control value (to stop watch time), and liquid test measurements, including estimating stopping points for "thinner" liquids.

Multiple samples were taken for statistically valid sets.

Two Compartment Tests

Pressure tests were configured to present flow rates for 25 and 50 psi on replacement liquids (water and 15/40 oil at room temperature).

Pressurized gas flow tests were not performed due to difficulty in arranging controlled experiments reflecting gas replacement under pressure.

Liquid Tests

Liquid flow rate tests reflected various system configurations including:

- Starting Vacuum 5 inch increments except for tests of near absolute vacuum (either 28 inches or 29 inches used)
- Hose Diameter and Length diameters varied between 3/16 inch and 1/4 inch
 ID; lengths up to 90 feet were tested
- Viscosity all material viscosities varied as a function of temperature
- Temperature 30° 180° F range tested; gasoline tested at room temperature only
- Height of Tank Placement above or below liquid

Flow rate test results are presented for the most meaningful interpretation of data. For example, oil tests showing flow rate data at 0° F are more difficult to interpret in terms of 0.0036 quarts per minute rather than 13 minutes for one quart.

Gaseous Tests

Gaseous tests were conducted on air and Freon using the following parameters:

- Starting vacuum
- Hose diameter
- Hose length
- Height of tank placement this parameter is not considered to impact this test due to low mass density of test gases

Outside the capabilities of test controls was qualitative information of Freon under pressures sufficient to cause a phase change to a liquid state.

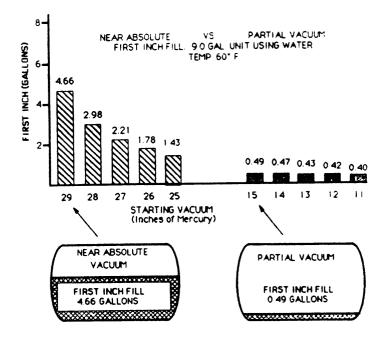
RESULTS AND DISCUSSION

PERFORMANCE RESULTS

Fill Capacity

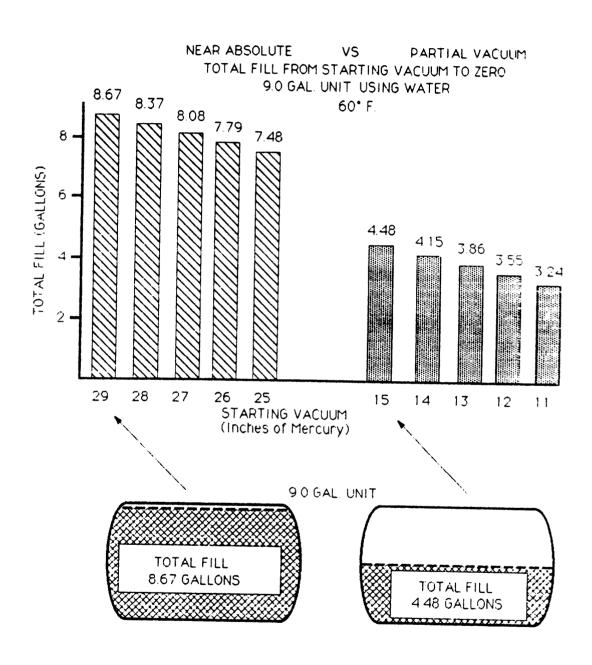
Tests results show a marked difference between fill capacity characteristics of a near absolute vacuum versus a partial vacuum for a given loss of vacuum. Although the amount of fill is proportional to starting vacuum, the amount for each inch (of mercury) of fill is significantly different (a factor of 10). Figure 3 illustrates the first inch fill capacity of a 9 gallon unit.

Figure 3. First Inch Fill Capacity: 9 Gallon Unit



Total fill is proportional to starting vacuum in most cases as seen in Figure 4.

Figure 4. Comparison of Fill to Starting Vacuum

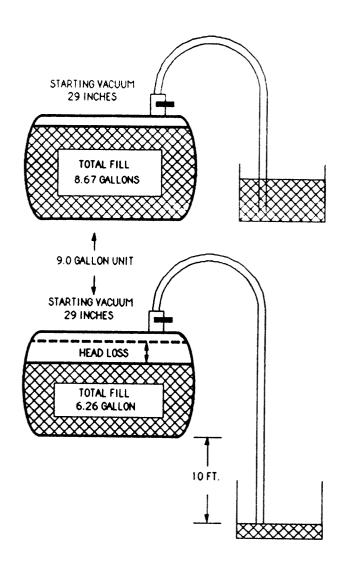


However, when operating with hot liquids, especially in the partial vacuum range, fills for the first inch (of mercury) are significantly lower than those at room temperatures. Both hot liquids and highly volatile liquids partially vaporize upon coming into contact with the vacuum, creating an immediate vacuum drop of several inches (under partial vacuum). For example, water showed a reduction of total fill from 4.48 gallons at 60° F to 3.16 gallons at 180° F at 15 inches vacuum -- a performance loss of 1.32 gallons.

At near absolute vacuum, hot liquid has a negligible effect on vacuum loss or capacity during the first few inches of fill as the actual percentage of air in the tank was almost nil.

"Head losses" are directly proportional to the elevation difference between the liquid and the vacuum as seen in Figure 5.

Figure 5. "Head" Loss Results



Flow Rates

Figure 6 represents the baseline data set using water as the "control". Flow rates, even for water, show a perceptible change as a function of viscosity and temperature. These flow rates incorporate the effects of temporary vacuum loss during the first few seconds of flow due to the liquid-to-vapor phase change.

Figure 6. Flow Rates: Water

FLOW RATES

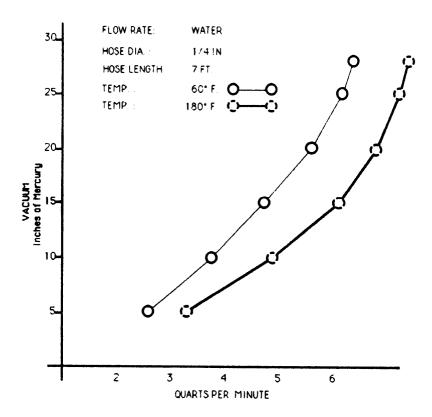
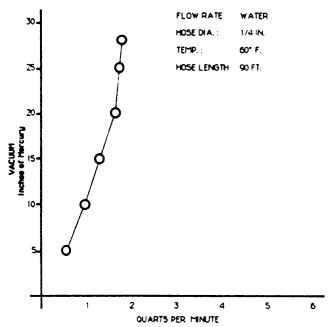


Figure 7 illustrates the frictional effects of an extended length of suction hose on flow rate. At near absolute vacuums, the flow rate is approximately 6 times faster through a short (7 feet) hose.

Figure 7. Frictional Effects of Suction Hose on Flow Rate



In larger diameter tubing, the flow is turbulent. Although extraction rates are very fast - water at 60° F: 1.6 gallons per minute (gpm) for a 1/4 inch diameter hose vs. 28.6 gpm for a 3/4 inch hose - a long, large diameter suction hose will significantly reduce the starting vacuum due to the air volume in the hose itself.

Flow rates for oil as a function of temperature and viscosity is seen in Figure 8:

Figure 8. Oil Flow Rates as a Function of Temperature and Viscosity

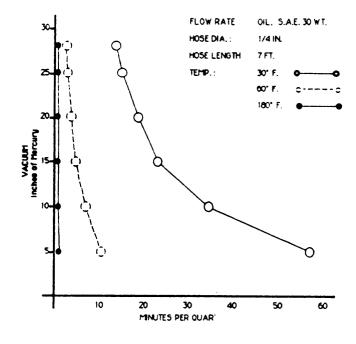


Figure 9 shows the relative flow rate relationships among test materials of extreme viscosities at 60° F.

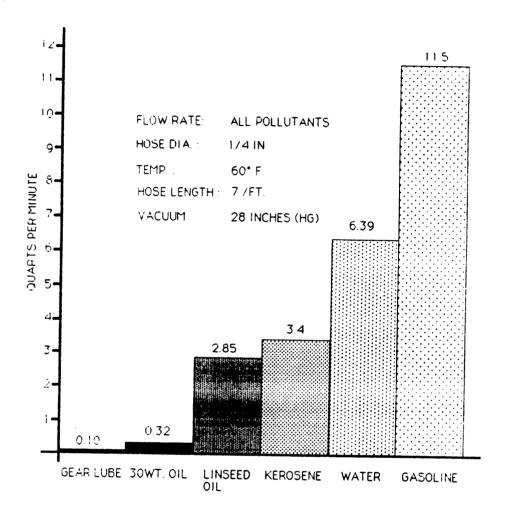
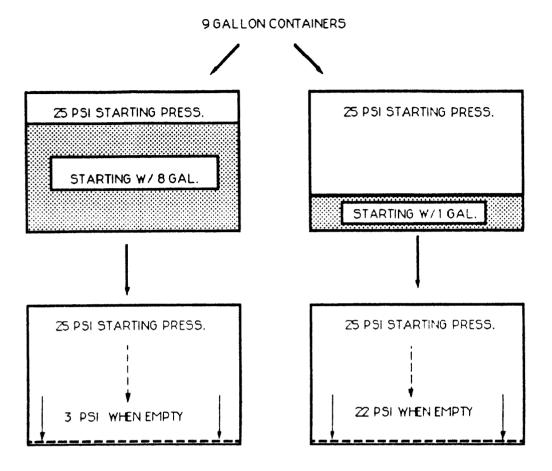


Figure 9. Flow Rates: All Test Materials

Pressure Return Results: Two-Compartment Unit

When the two-compartment model is used to remove waste oil with a vacuum and replace clean oil with pressure, one must start with adequate pressure to still have useful pressure near the end of the operation. Since the replacement oil is most likely at room temperatures, it will follow the same flow rates under equivalent pressure as represented by the previously shown vacuum flow rates. As illustrated in Figure 10, the amount of useful pressure remaining is dependent on the volume of starting liquid as well as the starting air pressure.

Figure 10. Useful Remaining Pressure as a Function of Starting Liquid Volume and Air Pressure



Worst Case Boundary Condition Tests

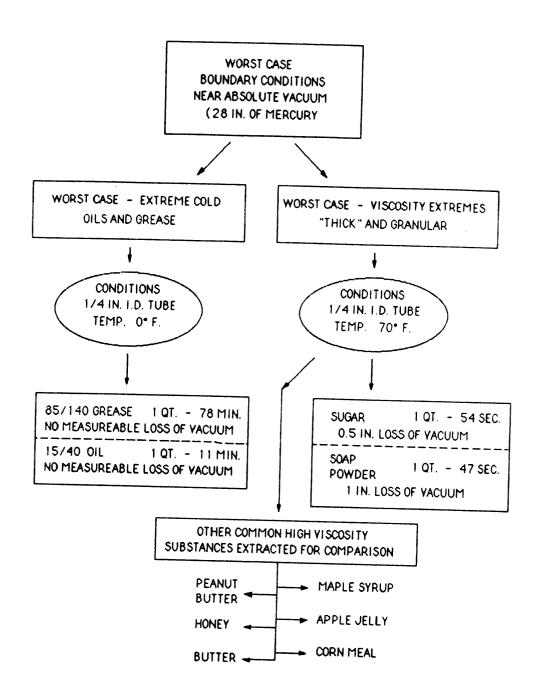
A variety of "worst case" or very thick liquids under very cold conditions were evaluated; these tests were performed qualitatively without any attempts to represent flow rate data. (See Figure 11.)

Tests were run using granular materials (powders, flour, corn meal, laundry detergent, etc.). Extraction of all materials proved successful.

The above listed samples were then mixed with water to test thick slurry feasibility. With the "pastier" mixtures (flour, corn meal), it was noted that the extraction hose became slightly "lined" with the slurry. If a higher proportion of water was added to the granular material, the hose was "washed down" with the vacuum, cleaning the hose.

Different materials with other than smooth liquid textures have properties that require specialized pick-up devices such as "sunflower heads". Also, powder and granular materials are not solid; their collection is necessarily accomplished with reduced volumetric efficiency.

Figure 11. Worst Case Boundary Conditions



Qualitative Conclusions on Vacuum Performance

- 1. The percentage fill of the container is directly proportional to the starting vacuum (for cold liquids).
- 2. The percentage fill of one inch of vacuum -- 28-27 inches vs. 15-14 inches -- is not the same.
- 3. The physical height of a vacuum above a liquid causes an effective loss of approximately one inch of vacuum per foot of elevation.
- 4. A liquid's viscosity has zero effect on the amount of vacuum used during the extraction process.
- 5. Hot engine oil (approximately 180° F) generally flows at approximately 1/2 gallon per minute in 1/4 inch ID tube. Oil at room temperature (approximately 65° F) slows to 1/2 gallon per 10 minutes using the same 1/4 inch ID tube.
- 6. Water at room temperature flows at approximately 1/2 gallon per minute through a 1/4 inch ID tube (roughly equal to hot engine oil).
- 7. Flow rate is approximately proportional to the square of the suction tube diameter.
- 8. A container charged to 29 inches of vacuum will fill to 97 percent of tank capacity.
- 9. A container charged to 15 inches of vacuum will fill to 51 percent of tank capacity.
- 10. The vacuum can only pull a liquid (near the weight of water) to a maximum elevation of approximately 33 feet.

Cost/Benefit Analysis

The savings using the Slurper™ vacuum technology are not so much in dollars as they are in volumetric reduction of improperly disposed waste oil. In the automotive field, "do-it-yourselfers" improperly dispose of approximately 120 million gallons of waste oil annually. If a Slurper™ rental/recycling process only saved 1 percent of this oil, 1.2 million gallons of oil would be recovered. Since one gallon of oil can contaminate one million gallons of drinking water, this 1 percent reduction could save 1.2 trillion gallons of water.

Numerous non-monetary factors enter into the technology application:

- Spill reduction from multiple transfers of waste oil.
- Increase in proper management of recovered material.
- Production and purchase cost savings for vacuum technology as compared to currently available commercial devices.

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Incentives

During the project, response to the containerized vacuum technique was extremely positive. Demonstrations and interfaces with businesses, private individuals, municipalities, and rescue squads generated the most positive feedback on the aspects of simplicity, containment, safety, and cleanliness of the operation.

Phase of Development

The technology is mature and in the marketplace. The present configuration is suitable for small quantity liquid applications under 8.5 gallons. Container size and shape can be modified to suit various applications. Inquiries have been received for up to 100 gallon models.

Accessories were developed to facilitate specific applications. One example is an extraction hose coupling which is fitted directly to the oil dipstick tube on properly configured marine engines.

Target Industry Potential

Immediate target markets range from "do-it-yourselfers" to small and medium sized businesses which handle lubricating oils. The approximate order of expected and projected acceptability and usage is:

- Marine
- Industrial
- Automotive
- Other oil related extraction process
- Commercial rental

The two-compartment prototype has multiple configurations making it adaptable to various materials-handling requirements.

Commercial rental of the Slurper™ has strong potential. In a "pilot" marine rental program, pleasure boaters and commercial fishermen paid \$10-\$15 (depending on rental size) to extract oil from their engines. The unit, full of oil, was then returned to the rental facility, and the oil was recycled.

A two-compartment Slurper™ could be rented as an "all-in-one" oil change kit. The unit could be charged with vacuum on one side to extract used oil and filled with new oil to replace in the engine.

Barriers

Container size (currently nine gallons capacity is the largest) is a limitation. There is a practical limit to the size of the container which must be transported when full of liquid. While the portable nature of the Slurper™ is a great convenience, some degree of conservatism must be exercised to optimize the containerized vacuum.

Misunderstandings as to the nature and use of vacuum, especially containerized vacuum, are common.

Potential Solutions

Understanding the engineering and non-engineering factors of vacuum techniques can solve the above problems. Availability of concise information and demonstration of portable, containerized vacuum principles will allow the Slurper™ to gain widest acceptance.

ENVIRONMENTALLY SAFE FOUNTAIN SOLUTIONS FOR THE PRINTING INDUSTRIES

by

David R. Johns Summit Resource Management, Inc. Fort Wayne, IN 46804

ABSTRACT

Summit Resource Management, Inc. formulated an ecologically compatible fountain solution for the printing industry. This new solution eliminated isopropyl alcohol and mineral acids. Ethylene glycol was also reduced through the substitution of propylene-based glycol. These reductions and substitutions decreased the levels of harmful vapors in the air and reduced the amount of toxins released into the printer's wastewater system.

INTRODUCTION

PROJECT DESCRIPTION

The off-set printing industry produces magazines, posters, brochures, cards, forms, and the like. An ingredient in the fountain solution used in the printing process is isopropyl alcohol (IPA), which is used to produce an etch that does not allow the transfer of ink to certain areas of the paper. Printers typically use 5 to 25 percent IPA mixed in the fountain solution. IPA generates harmful vapors, and some amount is generally disposed through the wastewater treatment system.

Summit Resource Management (Summit) formulated an alcohol-free, environmentally safe fountain solution. The definition "environmentally safe" had to meet the wastewater disposal requirements and strive for a vapor pressure level of zero. One goal was to eliminate such ingredients as ethylene glycol, glycol ether EB, heavy metals, mineral acids, phosphates, alcohols, and low boiling solvents.

After defining the need for certain properties in the traditional fountain solution, Summit searched for environmentally friendly ingredients that would provide the same function. Such performance functions include surface tension, viscosity, cleaning capability, compatibility with the inks, pH needs, and vapor and odor concerns.

<u>Unique Product Features/Advantages</u>

The reformulated fountain solution is made with food grade products that are available from domestic manufacturers.

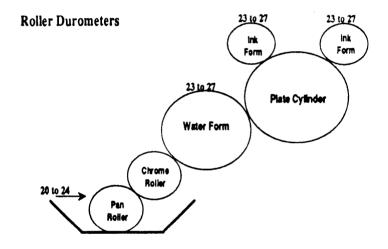
Summit Resource Management developed two fountain solution formulas: one for offset web presses with high speed printing, the other for sheet-fed presses that

print with slower speeds but higher quality. Both formulas have the same ingredients, but at different ratios to respond to different printing conditions. The products have a pleasant odor, are environmentally safe, and perform well. The products go into solution easily, making it simple to mix and clean up. Buffering agents stabilize the pH differences found in municipal water supplies. A softening agent was also added to balance the demands of hard and soft water; when using very hard water, all that is required is a 1 to 2 ounce increase in product concentration.

Process Schematic

Figure 1 shows a printing roller configuration and the pan where the fountain solution is introduced on the press. (Other variations include spraying the fountain solution on the water form roller and brushing the water solution mixture over the chrome roller.) The rollers generally are made from rubber, and the numbers on the diagram indicate the softness, or durometer, or the roller. A low number indicates a soft roller; a hard roller is numbered over 30. A soft roller provides the best results with the Summit product.

Figure 1. Roller Configuration and Fountain Solution Pan



APPLICATION

Process and Product Replaced

Summit's alcohol-free product replaces traditional fountain solutions plus alcohol. The pressman only needs to work with one product instead of two.

The required set-up process for using the Summit product is the opposite from the traditional process used by most press operators. When using the Summit solution, the pressman must start with a clean press, maintaining that cleanliness throughout the printing process. The pressman must then turn off the ink dials and set the water dials so that water covers the rollers. Once the rollers are clean the ink rollers are adjusted to the desired ink density. Traditional practices call for adjusting the water dials to the amount of ink.

Wastes Prevented

The Summit product allows the pressman to run a clean operation. Ink is reduced by 10 to 40 percent, and if set up correctly, the excess ink around the presses is eliminated. The wastewater is also cleaner.

Not only are environmental and health benefits realized through the elimination of alcohol, process improvements can also be noted. Alcohol attacks the inks and actually reduces the effective level of the ink. Alcohol eventually hardens or glazes the rubber rollers, preventing the transfer of solution to the rollers. Once these rollers are hard, they must be rebuilt or replaced.

PROCEDURE

DEMONSTRATION PROCEDURE

Summit developed an initial alcohol-free formula and tested samples for volatile organic compounds (VOCs) and vapor density. They then worked with several small printers to press-test the product.

Early results were mixed. In most cases, the product gave comparable results to traditional solution, but problems developed after a time:

- When the press stopped, the printing plate would oxidize, which then had to be cleaned.
- The new solution was more sensitive to changes and would not compensate for the use of excess ink.

Summit readjusted the formulas by adding gum (thickener) to deposit a coating on the plate to prevent oxidizing. The formula was also rebalanced to increase cleaning.

Once the formulation was refined, fifteen trial printing jobs were conducted at various printers. When conducting a trial, each pressman ran his own equipment under normal conditions. All colors were assessed from one-color to six-color presses.

The Summit fountain solution requires the rollers and blankets to be thoroughly clean. Once the press is clean, 4 ounces of solution is added to a gallon of water and placed in the water circulator. The ink dial is set to the lowest level. The water roller is set to completely wet the rollers. The ink rollers then are started and adjusted to the desired ink density; the ink setting will be lower than when using traditional products.

Four press trials are described in Table 1.

TABLE 1. Press Trial Parameters

PARAMETER	TRIAL 1	TRIAL 2	TRIAL 3	TRIAL 4
Equipment	Two-color Hiedelberg, 36-inch press; currently running 15% IPA solution	Harris four- color web, slow speed (600 feet per minute)	Kohl & Madden sheet fed, 36-inch press, 2 color	Four-color Komari
Customer	Off-Set One, Fort Wayne, IN	James River, Kendallville, IN	Robert Williams Graphics, NY	Container Corporation, Fort Wayne, IN
Colors	Red and black	Red, yellow, green, black	Red, blue	Red, yellow, blue, black
Paper	Sixty pound coated sheet fed	Boxboard (cereal box material)	Recycled bond paper	40 pound coated
Job	5,000 brochures	Cereal boxes	Forms and information sheets	Outside packaging for baseball cards
Concentration of Summit Product	4 ounces per gallon of city tap water	3 ounces per gallon of city well water	Started at 4 ounces per gallon of city water; increased to 5 ounces after the run was started	4 ounces per gallon of water
Water pH	10.5	9.0	7.8	10
Water pH After Addition of Fountain Solution	4.5	4.6	4.7	4.8
Roller Durometer	29 on ink roller	25	25	29
Roller Wash	Mineral spirit- based (not water soluble)	Mineral spirit- based	Two-part, water-based	Aromatic 100- base petroleum

Procedure	The press operator cleaned the rollers and old fountain solution from the equipment. He followed Summit's instructions in setting up the press. The pressman was concerned	The press operator cleaned and prepared the press according to Summit procedures.	The pressman cleaned the old ink and fountain solution from the press and ran the Summit solution according to instructions.	Between shifts, press operators quickly cleaned out old fountain solutions and wiped the ink rollers. The pressmen manually mixed the Summit solution and
	because the ink dials were set quite low.			solution and followed procedural instructions.

EVALUATION PARAMETERS

The parameters for evaluating the success of the Summit product were the same as for current products: dry time, dot gain, press speed, toning, scumming, number of impressions before cleaning or down-time problems, and slurring. Summit also focused on temperature and humidity levels, as changes in these two factors affect plate oxidation levels and ink acceptance.

Figure 2 is a Graphic Arts Technical Foundation chart for normal and non-acceptable printing that was used to judge the final printed product.

Figure 2. Normal and Non-acceptable Printing Standards



Samples of the Summit product were tested by an independent laboratory for VOCs and vapor pressure.

Summit established the following guidelines for pressmen using the Summit solution, as the new procedures differed from traditional operating practices.

- 1. Mix the Summit solution, 4 ounces per gallon of water.
- 2. Clean the rollers and check their durometer (softness); softer is preferred.
- 3. Set the ink dial to the lowest markings. Set the water dial low, but ensure the water roller is covered with the water/fountain solution mixture.
- 4. Start the press and adjust the ink density to the desired levels.

The success or failure of the print quality was based on the skill of each press operator, who would evaluate his work using such questions as:

- Were the dots that made up the actual print clean?
- Were the edges sharp?
- Were the colors the correct density and shade?
- Did the inks dry fast enough so as not to smear?
- Is the printed material saleable?

ASSESSMENT SUMMARY

Trial and error in formula development while working with the various pressmen was critical to the success of the project. For example, Summit found that no two presses performed alike, even if from the same manufacturer. Press operators tend to customize their equipment to their liking.

COST OF DEMONSTRATION

The total demonstration cost was \$46,781, with \$22,450 contributed by EPA through the Pollution Prevention By and For Small Business program, and the balance provided by Summit.

RESULTS AND DISCUSSION

PERFORMANCE RESULTS

Summit developed two products instead of the planned one, as a single product could not handle different speeds of the presses. The basic ingredients of the two products are the same, but are combined in different ratios. Summit learned that when the presses ran faster -- such as with web presses -- smaller amounts of the etch must remain on the rollers. Summit needed to increase the cleaning capability of the solution, leaving the correct viscosity of solution on the plate. The concentration of the product simply could not be diluted because the viscosity of the water on the rollers was not high enough to cover non-print areas. In comparison, alcohol is consistent in viscosity at a specific rate. A ten percent level of IPA always yields the same measurements, even when the fountain solution is dirty or weak in etch concentrations.

Summit altered the polymer level and increased solvency of their product to accommodate the differences of a clean fountain and a dirty one.

Summit also found that measuring the concentration of the fountain solution was unrelated to solution conductivity. Traditional fountain solutions contain small amounts of salts and metals that allow a conductivity meter to measure levels of fountain solution in the pan. As the concentration of solution drops, a mixer automatically adds more fountain solution. Summit solution additions are based on volume only, and Summit had to educated press operators that conductivity is not a key factor in the performance of a fountain solution.

PRODUCT QUALITY VARIANCE

Summit found little quality variance in the production of their new fountain solution. The project is a pleasant smelling and looking material, is easy to clean, and does not irritate the skin. The product was designed as a concentrate, with a recommended dilution of 4 ounces per gallon of water (compared to traditional product dilutions of 6 to 12 ounces per gallon of water).

CONDITIONS THAT IMPACT PERFORMANCE

Using the Summit product requires control changes in the ink and water levels as well as close monitoring of the press. The Summit solution requires a consistently clean press. When alcohol is used, the press operator does not have to be concerned with press cleanliness until the print job is finished. Printers must maintain a high volume operation, and Summit found that printers always were not willing to patiently fine tune the ink and water levels for the new solution or to clean the rollers sufficiently.

High temperature and humidity were initially troublesome, creating oxidation of the plates, but the final product formulation solved the problem. Conducting the press trials in the winter with low temperature and humidity -- ideal printing conditions -- made it challenging to judge the effects of these two variables.

Measuring the number of ounces of fountain solution was difficult for some larger printers that use conductivity meters to determine the proper time to add solution. Their systems were automatic and could not measure volume. Automatic fountain solution blending systems measure conductivity or acidic levels. The Summit solution does not contain metals, and the acid is buffered so that it will not react to minor changes.

Summit developed trouble shooting guidelines for press operators, as shown below in Table 2.

TABLE 2. Trouble Shooting Guidelinws for Summit Solution 6130X

PROBLEM	PROBABLE CAUSE	CORRECTIVE ACTION
Scumming - non-image	Running excess ink	Reduce ink level
area of plate accepting ink	Glazed offset blanket	Clean all gum, spray, etc. from the blanket
	Plate or press condition	Wet hone plate in problem area; if honed area continues to take ink, scumming is caused by an inking problem on press; if area stays clean, scumming is caused by plate sensitivity
	Excessive printing pressure	Consult appropriate press manual; pack to print
Tinting/Toning - fine ink particles adhere to non-image area of plate and	Ink emulsification	Replace with fresh ink and reduce water dampening
appear on sheet as a tint	Improper ink and/or dampening roller setting	Check setting on both ink and dampener rollers; adjust if necessary
Ink Roller Stripping - ink rollers do not accept ink	Running excessive water	Adjust ink/water balance, pH = 4.4 or above
	Glazed or worn roller	Clean or replace roller
	Fountain solution too concentrated, pH too low	Decrease concentration
Poor Ink Drying - ink does not dry	Fountain solution too acid	Check pH, reduced fountain solution causing set-off
	Running excessive ink	Reduce ink levels in delivery
	Water too high	Adjust water level
Plate Blinding - plate does not accept ink in all	Fountain solution too acid	Check pH, may have to adjust concentration
areas, or has low print contrast	Running excessive water	Adjust ink/water balance
	Cleaners have dried on plate	Wet-wash plate to remove cleaners

Improper Ink/Water Balance - difficulty in	Running excess water	Reduce dampener settings
maintaining print quality due to build-up, piling, or emulsification	Running excess ink	Reduce ink roller settings; if ink levels are low, may have to increase solution by half-ounce increments
Plate Wear - plate breaks	Running excess water	Reduce ink roller setting
down or sharpens prematurely	Fountain solution pH	Check pH and adjust if necessary

TABULATION OF DATA

Some information from the press trials were subjective, as the press operators opinions on product quality indicated success or failure of the test.

The Summit product worked well with all equipment tested except for an AB Dick press with a silver plate or paperback plate.

Table 3 shows a comparison of vapor pressure (the amount of evaporation over time) for traditional fountain solution and the Summit solution. Vapor pressure, along with related VOC content, has been reduced drastically. Traditional solutions are made with butyl cellosolve and other alcohols that are volatile. A typical concentrated solution contains approximately 5 pounds of VOCs per gallon. The Summit solution tested below detection levels for all VOCs (using guidelines from Test Method for Evaluating Solid Waste, Physical/Chemical Methods, Reference 5, Citation 8240, USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460, SW-846 Third Edition, November 1986).

TABLE 3. Vapor Pressire* Comparison of Traditional Fountain Solution and Summit Fountain Solution, and Water

Traditional Fountain Solution	Summit Fountain Solution	Water
56 - 108 mm Hg	< 2.5 mm Hg	16 mm Hg

^{*}Determined using a mercury manometer. Solutions were equilibrated to 20°C for 15 minutes before making the readings.

Press test results for the four previously described trials are shown in Table 4.

TABLE 4. Press Test Results

PARAMETER	TRIAL 1	TRIAL 2	TRIAL 3	TRIAL 4
Density	Brighter, more gloss	Good clear, high gloss	High gloss, sharp colors, good printing	Good, sharp colors, high gloss
Dot Gain (acceptable is <22%)	Less than 18%	21-22%	19%	18%
Dot Structure	Clean, clear, circular dots	Good, clean, sharp edges	Clean, clear dot, sharp edges	Clean, sharp edges; good circular structure
Ink Usage	Used 50% less black ink and 20% less red ink than the last time the job was run	Used 20% less black ink; 10% less red, yellow, and green inks	Reduced 8- 10%	Reduced 15- 20% depending on color
Set-up Usage	Used 50 sheets to adjust press & density; past set-ups were approx. 35-40 sheets	Achieved proper density within 300-350 feet	Ran 45 sheets before density & cleaning were correct	60 sheets (rollers were not cleaned well)
Cleaning	Press required more initial cleanup, but once clean, was able to maintain	Fountain solution started clean, but the ink would feed back onto the roller, scumming on the boxboard after 10,000 feet. Although the roller was cleaned, the scum would return.	Increasing concentration of red ink corrected initial problems	Once rollers were cleaned several times, cleanliness was maintained
Dry Time	Sheet dried faster and could be cut & folded sooner	Faster	Best seen by pressman	Improved

Summit Resource Management conducted fifteen trials with similar success. Customers who continued to use the Summit product had problems if they assumed the product would work "on its own" and did not work within the sensitivity parameters of the product.

Test result variances did not seem to follow any predictable trends. Local conditions, such as water hardness, press subtleties as fine-tuned by an operator, and pressman skill and attention to detail influenced the quality of the printed product.

In general, the printers liked the Summit product. Quality was better, gloss was greater, dot gain was less, drying time was less, and ink use was reduced. The same or better quality product could be printed without alcohol or toxic ingredients found in traditional fountain solutions.

Cost/Benefit Analysis

The initial cost to purchase one gallon of the Summit fountain solution is approximately twice as much as for traditional products. However, using the Summit product realizes a savings when the other costs of using IPA are considered, as shown in Table 5.

TABLE 5. Cost Comparison of Summit Fountain Solution and Traditional Product

	Summit Fountain Solution	Traditional Fountain Solution and IPA
Costs		
Fountain Solution	\$25/gallon	\$12/gallon
Black Ink	\$3/pound	\$3/pound
Alcohol	\$0	\$2.50/gallon
Requirements per Total Gallo	n of Mixed Fountain Soluti	on
Ounces per Gallon of Water	4	6
Ounces of Alcohol	0	19 (15%)
Ounces of Ink	1.4	2
Savings Review		
Material Cost	\$0.78	\$0.56
Alcohol	\$0	\$0.375
Ink	\$0.28	\$0.375
Total Use Cost	\$1.04	\$1.31

These savings do not include hazardous waste disposal or water treatment costs associated with the use of traditional solutions.

CONCLUSIONS

POLLUTION PREVENTION ASSESSMENT

Incentives

The Summit fountain solution reduces pollution by eliminating IPA and heavy metals found in traditional fountain solutions. When correctly used, the Summit product enables the printer to obtain equal or better results in the quality of the printed product.

Summit fountain solution also reduces the need for wastewater treatment plants especially if used with less hazardous inks. For example, the Summit solution has been tested successfully with soy inks.

Barriers

Press operators must modify their procedures to successfully integrate the Summit solution into their process.

New "dry plate" technology is currently being developed that would eliminate the use of fountain solutions. Although printing quality is excellent using this new process, costs are high, and new presses are required.